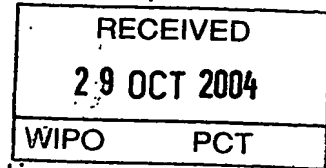




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Die angehefteten Unterlagen stimmen mit der ursprünglich eingereichten Fassung der auf dem nächsten Blatt bezeichneten europäischen Patentanmeldung überein.

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Les documents fixés à cette attestation sont conformes à la version initialement déposée de la demande de brevet européen spécifiée à la page suivante.

Patentanmeldung Nr. Patent application No. Demande de brevet n°

03103559.5

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(Falls die Bezeichnung der Erfindung nicht angegeben ist, siehe Beschreibung.
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Molecular weight increase and modification of polycondensates

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Molecular Weight Increase and Modification of Polycondensates

The present invention relates to a process for increasing the molecular weight and for the modification of polycondensates. Further aspects are the use of an additive blend effecting the increase in molecular weight without imparting color to the polycondensates as well as the polycondensates obtainable by that process.

Polycondensates, for example polyamides, polycarbonates or polyesters, in particular polyethylene terephthalate (PET) and polybutylene terephthalate (PBT) as well as polyester copolymers and polyester blends e.g. with polycarbonate (PBT/PC), are important thermoplastics belonging to the group of the engineering plastics. Partially crystalline polyesters are used for injection moulding compounds and are distinguished by high strength and rigidity, high dimensional stability and favourable wear characteristics. Amorphous polyesters have high transparency, superior toughness and excellent stress cracking resistance and are processed, for example, to hollow articles. Another field of application of PET is the production of fibres and foils.

The mechanical and physical properties depend essentially on the molecular weight of the polymer. Polycondensates are prepared by condensation in the melt. Average molecular weights can thus be obtained. For some applications, for example drinks packs and technical fibres, higher molecular weights are necessary. These can be obtained by solid phase polycondensation (S.Fakirov, *Kunststoffe*, 74 (1984), 218 and R. E. Grützner, A. Koine, *Kunststoffe*, 82 (1992), 284). The prepolymer is in this case subjected to thermal treatment above the glass transition temperature and below the melt temperature of the polymer under inert gas or under vacuum. However, this method is very time- and energy-consuming. Increasing the intrinsic viscosity requires a residence time of up to 12 hours under vacuum or under inert gas at temperatures from 180 to 240°C.

Another possibility for obtaining higher molecular weights of polycondensates and, in particular, of polyesters is to add a tetracarboxylic acid anhydride and a sterically hindered hydroxyphenylalkylphosphonate to the polycondensate and process the mixture in the melt. This is, for example, described in US 5,693,681.

Furthermore WO 96/34909 and WO 98/47940 disclose the use of bis-acyllactams for increasing the molecular weight of polycondensates, in particular of polyamide. These products, which are, for example, sold by DSM under the trade name Allinco® compensate

the hydrolytic/thermal degradation during melt processing of polycondensates. However, these products have a strong influence on the color of the processed polycondensates. In addition the reaction is rather slow and does not ideally fit into typical process cycle times.

- 5 It has now been found that the addition of a combination of a bis- acyllactame a phosphite, phosphinate or phosphonate and/or a benzofuran-2-one type compound to a polycondensate, with subsequent reactive extrusion of the mixture, allows to substantially increase the molecular weight within short reaction times, without imparting color to the extruded article.
- 10 This is of particular interest since the high effort of solid state polycondensation can be avoided. The desired higher molecular weight of the polycondensates can be achieved by applying the instant compositions and processes within a melt processing step (e.g. reactive extrusion), which is much less effort than a solid state polycondensation. Furthermore, the instant process provides the flexibility to the converters to adjust the molecular weight of the
- 15 polycondensates, according exactly to their needs. Yet another advantage is that the thermal/hydrolytic degradation of polycondensates during melt-processing is prevented or at least significantly reduced.

- This is also advantageous in the case of used or thermally or hydrolytically damaged
- 20 polycondensates, where the damage typically goes hand in hand with a decrease of the molecular weight.

- By means of the process of this invention it is possible to increase the molecular weight also in the case of polycondensate recyclates from useful material collections, such as used
- 25 packages (foils and bottles) and waste textiles. Recyclates can then be used for high-quality recycling, for example in the form of high-performance fibres, injection moulding articles, in extrusion applications or in the form of foams. Such recyclates originate, for example, also from industrial or domestic useful material collections, from production wastes, such as from fibre production and trimmings, or from obligatory returnables, such as bottle collections of
- 30 PET drinks packs.

- In addition, the physicochemical properties are altered through the process of this invention such, that polycondensates can be foamed or extrusion blow moulded into films and containers and other hollow articles.

One aspect of the invention is a process for increasing the molecular weight and/or for the modification of a polycondensate, which process comprises adding to the polycondensate

a) at least one bis-acyllactam;

b1) at least one phosphite, phosphinate or phosphonate; or

5 b2) at least one benzofuran-2-one type compound or

b3) at least one phosphite, phosphinate or phosphonate and one benzofuran-2-one type compound

and processing the mixture in the melt.

10 Phosphonates are in general preferred.

In addition to polyester, polyamide or polycarbonate, this invention also embraces the corresponding copolymers and blends, for example PBT/PS, PBT/ASA, PBT/ABS, PBT/PC, PET/ABS, PET/PC, PBT/PET/PC, PBT/PET, PA/PP, PA/PE and PA/ABS. However, it needs to be taken into account that the novel process, like all methods allowing exchange reactions
15 between the components of the blend, may influence the blends, i.e. may result in the formation of copolymeric structures.

A preferred process is that wherein the polycondensate is an aliphatic or aromatic polyester, an aliphatic or aromatic polyamide or polycarbonate, or a blend or copolymer thereof.

20

The polycondensate is for example polyethylene terephthalate (PET), polybutylene terephthalate (PBT), polyethylenenaphthalate (PEN), polytrimethylene terephthalate (PTT), a copolyester, PA 6, PA 6.6, a polycarbonate containing bisphenol A, bisphenol Z or bisphenol F linked via carbonate groups.

25

Preferred polycondensates are PET, a copolymer with PET or PBT.

Polyamides, i.e. both virgin polyamides and polyamide recyclates, are understood to be, for example, aliphatic and aromatic polyamides or copolyamides which are derived from diamines and dicarboxylic acids and/or of aminocarboxylic acid or the corresponding lactams. Suitable polyamides are for example: PA 6, PA 11, PA 12, PA 46, PA 66, PA 69, PA 610, PA 612, PA 10.12, PA 12.12 and also amorphous polyamides and thermoplastic polyamide elastomers such as polyether amides of the Vestamid, Grilamid ELY60, Pebax, Nyim and Grilon ELX type. Polyamides of the cited type are commonly known and are commercially
35 available.

The polyamides used are preferably crystalline or partially crystalline polyamides and, in particular, PA6 and PA6.6 or their blends, as well as recyclates on this basis, or copolymers thereof.

5

The polyesters, i.e. virgin polyester as well as polyester recyclate, may be homopolyesters or copolyesters which are composed of aliphatic, cycloaliphatic or aromatic dicarboxylic acids and diols or hydroxycarboxylic acids.

- 10 The polyesters can be prepared by direct esterification (PTA process) and also by transesterification (DMT process). Any of the known catalyst systems may be used for the preparation.

- 15 The aliphatic dicarboxylic acids can contain 2 to 40 carbon atoms, the cycloaliphatic dicarboxylic acids 6 to 10 carbon atoms, the aromatic dicarboxylic acids 8 to 14 carbon atoms, the aliphatic hydroxycarboxylic acids 2 to 12 carbon atoms and the aromatic and cycloaliphatic hydroxycarboxylic acids 7 to 14 carbon atoms.

- 20 The aliphatic diols can contain 2 to 12 carbon atoms, the cycloaliphatic diol 5 to 8 carbon atoms and the aromatic diols 6 to 16 carbon atoms.

Polyoxyalkylene glycols having molecular weights from 150 to 40000 may also be used.

- 25 Aromatic diols are those in which two hydroxyl groups are bound to one or to different aromatic hydrocarbon radicals.

- 30 It is also possible that the polyesters are branched with small amounts, e.g. from 0.1 to 3 mol%, based on the dicarboxylic acids present, of more than difunctional monomers (e.g. pentaerythritol, trimellitic acid, 1,3,5-tri(hydroxyphenyl)benzene, 2,4-dihydroxybenzoic acid or 2-(4-hydroxyphenyl)-2-(2,4-dihydroxyphenyl)propane).

Suitable dicarboxylic acids are linear and branched saturated aliphatic dicarboxylic acids, aromatic dicarboxylic acids and cycloaliphatic dicarboxylic acids.

- 35 Suitable aliphatic dicarboxylic acids are those containing 2 to 40 carbon atoms, for example oxalic acid, malonic acid, dimethylmalonic acid, succinic acid, pimelic acid, adipic acid, trimethyladipic acid, sebacic acid, azelaic acid and dimeric acids (dimerization products of

unsaturated aliphatic carboxylic acids such as oleic acid), alkylated malonic and succinic acids such as octadecylsuccinic acid.

5 Suitable cycloaliphatic dicarboxylic acids are: 1,3-cyclobutanedicarboxylic acid, 1,3-cyclopentanedicarboxylic acid, 1,3- and 1,4-cyclohexanedicarboxylic acid, 1,3- and 1,4-(dicarboxymethyl)cyclohexane, 4,4'-dicyclohexyldicarboxylic acid.

10 Suitable aromatic dicarboxylic acids are: In particular terephthalic acid, isophthalic acid, o-phthalic acid, and 1,3-, 1,4-, 2,6- or 2,7-naphthalenedicarboxylic acid, 4,4'-diphenyldicarboxylic acid, 4,4'-diphenylsulfonedicarboxylic acid, 4,4'-benzophenonedicarboxylic acid, 1,1,3-trimethyl-5-carboxyl-3-(p-carboxylphenyl)indane, 4,4'-diphenyl ether dicarboxylic acid, bis-p-(carboxylphenyl)methane or bis-p-(carboxylphenyl)ethane.

15 The aromatic dicarboxylic acids are preferred, in particular terephthalic acid, isophthalic acid and 2,6-naphthalenedicarboxylic acid.

20 Other suitable dicarboxylic acids are those containing -CO-NH-groups; they are described in DE-A2414349. Dicarboxylic acids containing N-heterocyclic rings are also suitable, for example those which are derived from carboxylalkylated, carboxylphenylated or carboxybenzylated monoamine-s-triazinedicarboxylic acids (viz. DE-A-2121184 and 2533675), mono- or bishydantoins, optionally halogenated benzimidazoles or parabanic acid. The carboxyalkyl group can in this case contain 3 to 20 carbon atoms.

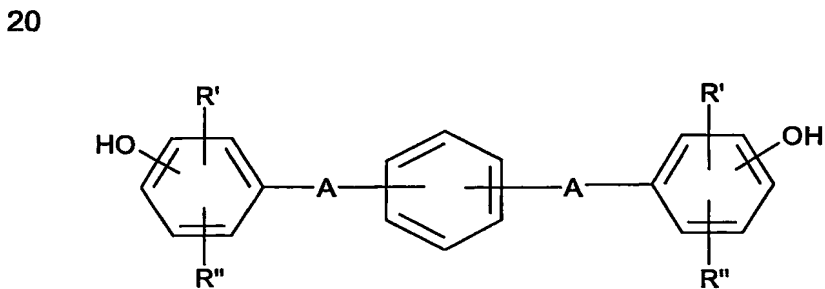
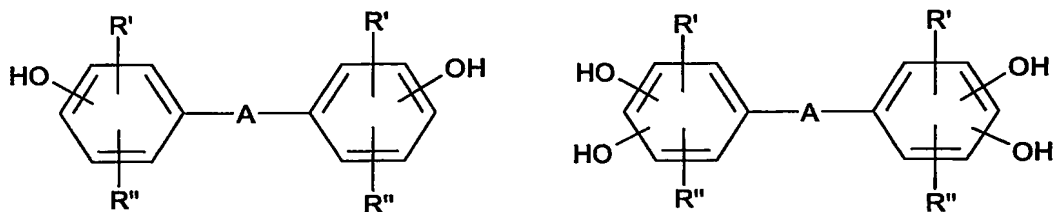
25 Suitable aliphatic diols are the linear and branched aliphatic glycols, in particular those containing 2 to 12, preferably 2 to 6, carbon atoms in the molecule, for example: ethylene glycol, 1,2- and 1,3-propylene glycol, 1,2-, 1,3-, 2,3- or 1,4-butanediol, pentyl glycol, neopentyl glycol, 1,6-hexanediol, 1,12-dodecanediol. A suitable cycloaliphatic diol is e.g. 1,4-dihydroxycyclohexane. Other suitable aliphatic diols are e.g. 1,4-bis(hydroxymethyl)cyclohexane, aromatic-aliphatic diols such as p-xylylene glycol or 2,5-dichloro-p-xylylene glycol, 2,2-(β-hydroxyethoxyphenyl)propane and also polyoxyalkylene glycols such as diethylene glycol, triethylene glycol, polyethylene glycol or polypropylene glycol. The alkylene diols are preferably linear and preferably contain 2 to 4 carbon atoms.

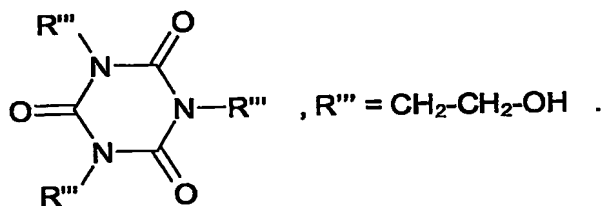
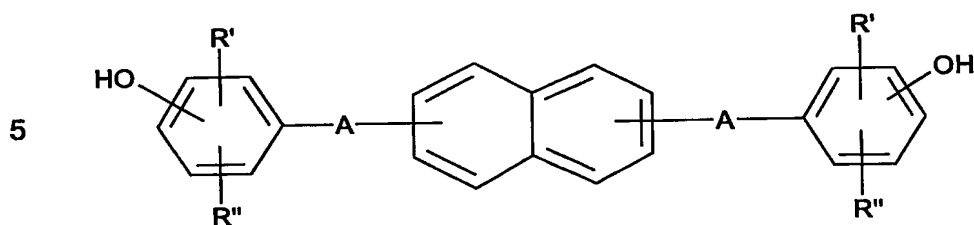
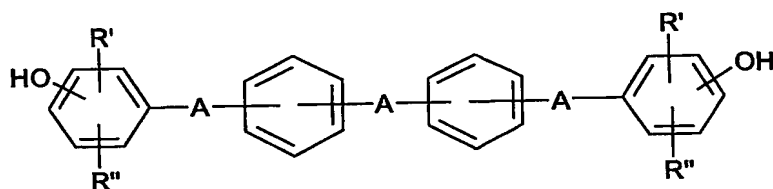
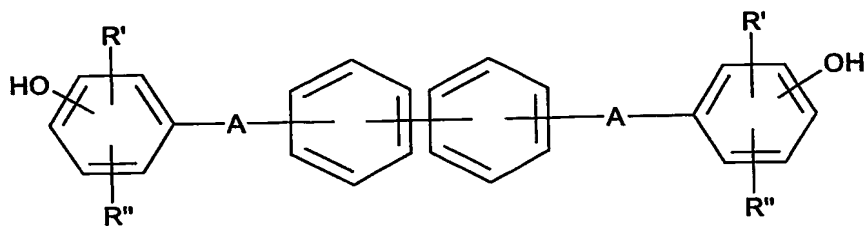
35 Preferred diols are the alkylenediols, 1,4-dihydroxycyclohexane and 1,4-bis(hydroxymethyl)cyclohexane. Particularly preferred are ethylene glycol, 1,4-butanediol and 1,2- and 1,3-propylene glycol.

Other suitable aliphatic diols are the β -hydroxyalkylated, in particular β -hydroxyethylated, bisphenols such as 2,2-bis[4'-(β -hydroxyethoxy)phenyl]propane. Other bisphenols will be mentioned later.

5 Another group of suitable aliphatic diols are the heterocyclic diols described in DE-A-1812003, DE-A-2342432, DE-A-2342372 and DE-A-2453326, for example: N,N'-bis(β -hydroxyethyl)-5,5-dimethylhydantoin, N,N'-bis(β -hydroxypropyl)-5,5-dimethylhydantoin, methylenebis[N-(β -hydroxyethyl)-5-methyl-5-ethylhydantoin], methylenebis[N-(β -hydroxyethyl)-
10 5,5-dimethylhydantoin], N,N'-bis(β -hydroxyethyl)benzimidazolone, N,N'-bis(β -hydroxyethyl)-(tetrachloro)benzimidazolone or N,N'-bis(β -hydroxyethyl)-(tetrabromo)benzimidazolone.

Suitable aromatic diols are mononuclear diphenols and, in particular dinuclear diphenols carrying a hydroxyl group at each aromatic nucleus. Aromatic will be taken to mean preferably hydrocarbonaromatic radicals, such as phenylene or naphthylene. Besides e.g. hydroquinone, resorcinol or 1,5-, 2,6- and 2,7-dihydroxynaphthalene, the bisphenols are to be
15 mentioned in particular, which can be represented by the following formulae:





10 The hydroxyl groups can be in m-position, preferably in p-position, and R' and R'' in these formulae can be alkyl containing 1 to 6 carbon atoms, halogen, such as chloro or bromo, and, in particular, hydrogen atoms. A may be a direct bond or -O-, -S-, -(O)S(O)-, -C(O)-, -P(O)(C₁-C₂₀alkyl)-, unsubstituted or substituted alkylidene, cycloalkylidene or alkylene.

15 Examples of unsubstituted or substituted alkylidene are: ethylidene, 1,1- or 2,2-propylidene, 2,2-butylidene, 1,1-isobutylidene, pentylidene, hexylidene, heptylidene, octylidene, dichloroethylidene, trichloroethylidene.

Examples of unsubstituted or substituted alkylene are methylene, ethylene, phenylmethylene, diphenylmethylene, methylphenylmethylene. Examples of cycloalkylidene are cyclopentylidene, cyclohexylidene, cycloheptylidene and cyclooctylidene.

- 5 Examples of bisphenols are: bis(p-hydroxyphenyl) ether or bis(p-hydroxyphenyl) thioether, bis(p-hydroxyphenyl)sulfone, bis(p-hydroxyphenyl)methane, bis(4-hydroxyphenyl)-2,2'-biphenyl, phenylhydroquinone, 1,2-bis(p-hydroxyphenyl)ethane, 1-phenylbis(p-hydroxyphenyl)ethane, diphenylbis(p-hydroxyphenyl)methane, diphenylbis(p-hydroxyphenyl)ethane, bis(3,5-dimethyl-4-hydroxyphenyl)sulfone, bis(3,5-dimethyl-4-hydroxyphenyl)-p-diisopropylbenzene, 10 bis(3,5-dimethyl-4-hydroxyphenyl)-m-diisopropylbenzene 2,2-bis(3',5'-dimethyl-4'-hydroxyphenyl)propane, 1,1- or 2,2-bis(p-hydroxyphenyl)butane, 2,2-bis(p-hydroxyphenyl)hexafluoropropane, 1,1-dichloro- or 1,1,1-trichloro-2,2-bis(p-hydroxyphenyl)ethane, 1,1-bis(p-hydroxyphenyl)cyclopentane and, in particular, 2,2-bis(p-hydroxyphenyl)propane (bisphenol A) and 1,1-bis(p-hydroxyphenyl)cyclohexane (bisphenol C).
- 15 Suitable polyesters of hydroxycarboxylic acids are, for example, polycaprolactone, polypivalolactone or the polyesters of 4-hydroxycyclohexanecarboxylic acid, 2-hydroxy-6-naphthalene carboxylic acid or 4-hydroxybenzoic acid.

20 Other suitable compounds are polymers which may predominantly contain ester bonds or also other bonds, for example polyester amides or polyester imides.

Polyesters containing aromatic dicarboxylic acids have become most important, in particular the polyalkylene terephthalates. Accordingly, those novel moulding compositions are preferred wherein the polyester consists to at least 30 mol%, preferably to at least 40 mol%, of aromatic dicarboxylic acids and to at least 30 mol%, preferably to at least 40 mol%, of alkylenediols containing preferably 2 to 12 carbon atoms, based on the polyester.

25

In this case the alkylenediol is, in particular, linear and contains 2 to 6 carbon atoms, for example ethylene glycol, tri-, tetra- or hexamethylene glycol and the aromatic dicarboxylic acid, terephthalic acid and/or isophthalic acid.

30

Particularly suitable polyesters are PET, PBT, PEN, PTT and corresponding copolymers, PET and its copolymer being especially preferred. The process is also particularly important in the case of PET recyclates originating, for example, from bottle collections such as collections of the beverages industry. These materials preferably consist of terephthalic acid, 2,6-naphthalenedicarboxylic acid and/or isophthalic acid in combination with ethylene glycol, 35 diethylene glycol and/or 1,4-bis(hydroxymethyl)propanediol.

Polyester blends to be mentioned in particular are those comprising polycarbonate.

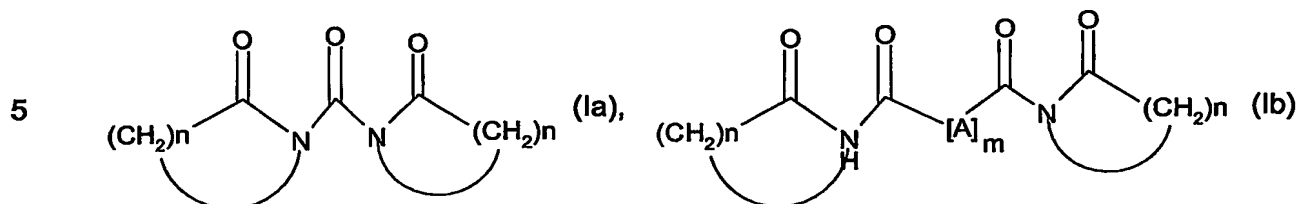
Polycarbonate (PC) is understood to mean both virgin polycarbonate and polycarbonate re-
5 cyclate. PC is obtained, for example, from bisphenol A and phosgene or phosgene analog
such as trichloromethylchloroformate, triphosgene or diphenylcarbonate, by condensation in
the latter case usually with addition of a suitable transesterification catalyst, for example a
boron hydride, an amine, such as 2-methylimidazole or a quaternary ammonium salt; in addi-
10 tion to bisphenol A other bisphenol components may also be used and it is also possible to
use halogenated monomers in the benzene nucleus. Particularly suitable bisphenol compo-
nents to be mentioned are: 2,2-bis(4'-hydroxyphenyl)propane (bisphenol A), 2,4'-dihydroxy-
diphenylmethane, bis(2-hydroxyphenyl)methane, bis(4-hydroxyphenyl)methane, bis(4-
hydroxy-5-propylphenyl)methane, 1,1-bis(4'-hydroxyphenyl)ethane, bis(4-hydroxyphenyl)-
cyclohexylmethane, 2,2-bis(4'-hydroxyphenyl)-1-phenylpropane, 2,2-bis(3',5'-dimethyl-4'-
15 hydroxyphenyl)propane, 2,2-bis(3',5'-dibromo-4'-hydroxyphenyl)propane, 2,2-bis(3',5'-dichlo-
ro-4'-hydroxyphenyl)propane, 1,1-bis(4'-hydroxyphenyl)cyclododecane, 1,1-bis(3',5'-dimethyl-
4'-hydroxyphenyl)cyclododecane, 1,1-bis(4'-hydroxyphenyl)-3,3,5-trimethylcyclohexane, 1,1-
bis(4'-hydroxyphenyl)-3,3,5,5-tetramethylcyclohexane, 1,1-bis(4'-hydroxyphenyl)-3,3,5-
20 trimethylcyclopentane and the bisphenols mentioned above. The polycarbonates may also
be branched by suitable amounts of more than difunctional monomers (examples as indi-
cated above for the polyesters).

The polyester copolymers or blends, which may be used in the novel process are prepared in
customary manner from the starting polymers. The polyester component is preferably PET,
25 PBT, and the PC component is preferably a PC based on bisphenol A. The ratio of polyester
to PC is preferably from 95:5 to 5:95, a particularly preferred ratio being that in which one
component makes up at least 75%.

This invention is also of interest in the case of polyester recyclates, such as are recovered
30 from production wastes, useful material collections or through so-called obligatory return-
ables e.g. from the beverage packaging industry, automotive industry or from the electronics
area. The polycondensate recyclates are in this case in many ways thermally and/or
hydrolytically damaged. These recyclates may additionally also contain subordinate amounts
of admixtures of plastics of different structure, for example polyolefins, polyurethanes, ABS
35 or PVC. Furthermore, these recyclates may also contain admixtures owing to standard

impurities, such as residues of colourants, adhesives, contact media or paints, traces of metal, water, operating agents, or inorganic salts.

The bis-acyllactam is for example of formula Ia or Ib



wherein A is C₁-C₁₈alkylen, C₂-C₁₈alkylene interrupted by at least one oxygen atom, C₁-C₁₈alkenylene, phenylene, phenylene-C₁-C₁₈alkylene, C₁-C₁₈alkylene-phenylene, or C₁-C₁₈alkylene-phenylene-C₁-C₁₈alkylene;

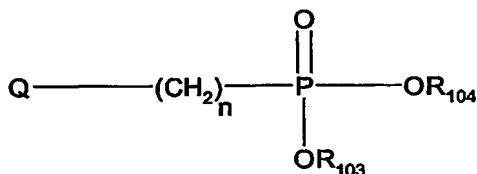
10 m is 0 or 1 and

n is a number from 3 to 12.

These compounds are known and partially items of commerce, for example under the trade name Allinco® from DSM. The compounds, their preparation and use is, for example, described in WO 96/34909 and WO 98/ 47940.

15

Preferably the phosphonate is of formula II



(II), wherein

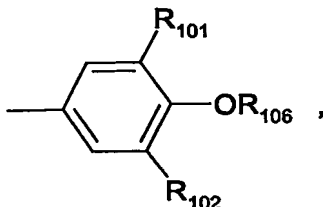
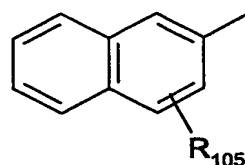
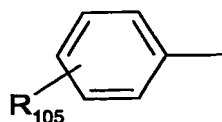
20 R₁₀₃ is H, C₁-C₂₀alkyl, unsubstituted or C₁-C₄alkyl-substituted phenyl or naphthyl, R₁₀₄ is hydrogen, C₁-C₂₀alkyl, unsubstituted or C₁-C₄alkyl-substituted phenyl or naphthyl; or M^{r+} / r,

M^{r+} is an r-valent metal cation or the ammonium ion,

n is 0, 1, 2, 3, 4, 5 or 6, and

25 r is 1, 2, 3 or 4;

Q is hydrogen, $-X-C(O)-OR_{107}$, or a radical



R_{101} is isopropyl, tert-butyl, cyclohexyl, or cyclohexyl which is substituted by 1-3 C_1-C_4 alkyl groups,

- 5 R_{102} is hydrogen, C_1-C_4 alkyl, cyclohexyl, or cyclohexyl which is substituted by 1-3 C_1-C_4 alkyl groups,

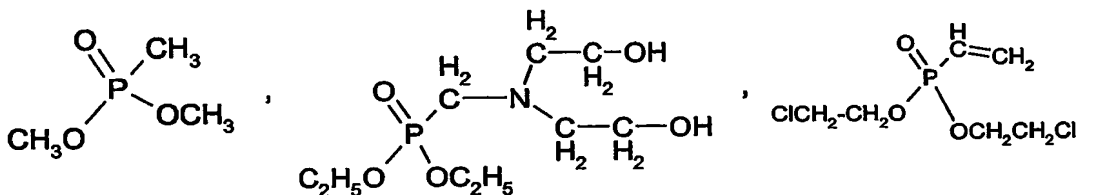
R_{105} is H, C_1-C_{18} alkyl, OH, halogen or C_3-C_7 cycloalkyl;

R_{106} is H, methyl, trimethylsilyl, benzyl, phenyl, sulfonyl or C_1-C_{18} alkyl;

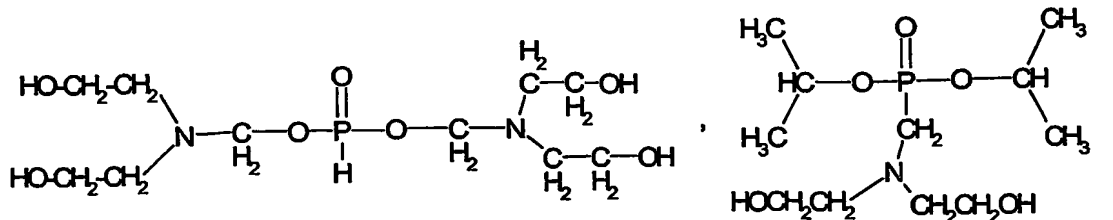
R_{107} is H, C_1-C_{10} alkyl or C_3-C_7 cycloalkyl; and

- 10 X is phenylene, C_1-C_4 alkyl group-substituted phenylene or cyclohexylene.

Other suitable phosphonates are listed below.



15





Sterically hindered hydroxyphenylalkylphosphonic acid esters or half-esters, such as those known from US 4 778 840, are preferred.

1000

Alkyl substituents containing up to 18 carbon atoms are suitably radicals such as methyl, ethyl, propyl, butyl, pentyl, hexyl and octyl, stearyl and also corresponding branched isomers; C₂-C₄alkyl and isooctyl are preferred.

15 C₁-C₄Alkyl-substituted phenyl or naphthyl which preferably contain 1 to 3, more preferably 1 or 2, alkyl groups is e.g. o-, m- or p-methylphenyl, 2,3-dimethylphenyl, 2,4-dimethylphenyl, 2,5-dimethylphenyl, 2,6-dimethylphenyl, 3,4-dimethylphenyl, 3,5-dimethylphenyl, 2-methyl-4-

ethylphenyl, 4-tert-butylphenyl, 2-ethylphenyl, 2,6-diethylphenyl, 1-methylnaphthyl, 2-methylnaphthyl, 4-methylnaphthyl, 1,6-dimethylnaphthyl or 4-tert-butyl-naphthyl.

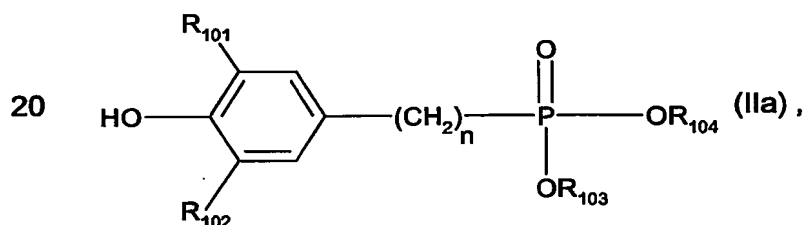
5 C₁-C₄Alkyl-substituted cyclohexyl which preferably contains 1 to 3, more preferably 1 or 2, branched or unbranched alkyl group radicals, is e.g. cyclopentyl, methylcyclopentyl, dimethylcyclopentyl, cyclohexyl, methylcyclohexyl, dimethylcyclohexyl, trimethylcyclohexyl or tert-butylcyclohexyl.

10 A mono-, di-, tri- or tetra-valent metal cation is preferably an alkali metal, alkaline earth metal, heavy metal or aluminium cation, for example Na⁺, K⁺, Mg⁺⁺, Ca⁺⁺, Ba⁺⁺, Zn⁺⁺, Al⁺⁺⁺, or Ti⁺⁺⁺⁺. Ca⁺⁺ is particularly preferred.

Preferred compounds of formula I are those containing at least one tert-butyl group as R₁ or R₂. Very particularly preferred compounds are those, wherein R₁ and R₂ are at the same time tert-butyl.

15 n is preferably 1 or 2 and, in particular 1.

For example the phosphonate is of formula IIa



wherein

R₁₀₁ is H, isopropyl, tert-butyl, cyclohexyl, or cyclohexyl which is substituted by 1-3 C₁-C₄alkyl groups,

25 R₁₀₂ is hydrogen, C₁-C₄alkyl, cyclohexyl, or cyclohexyl which is substituted by 1-3 C₁-C₄alkyl groups,

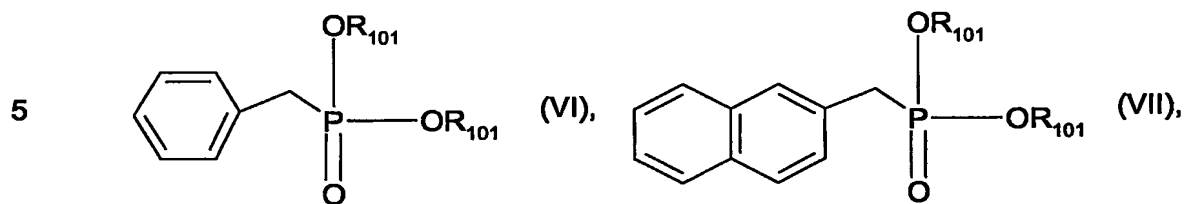
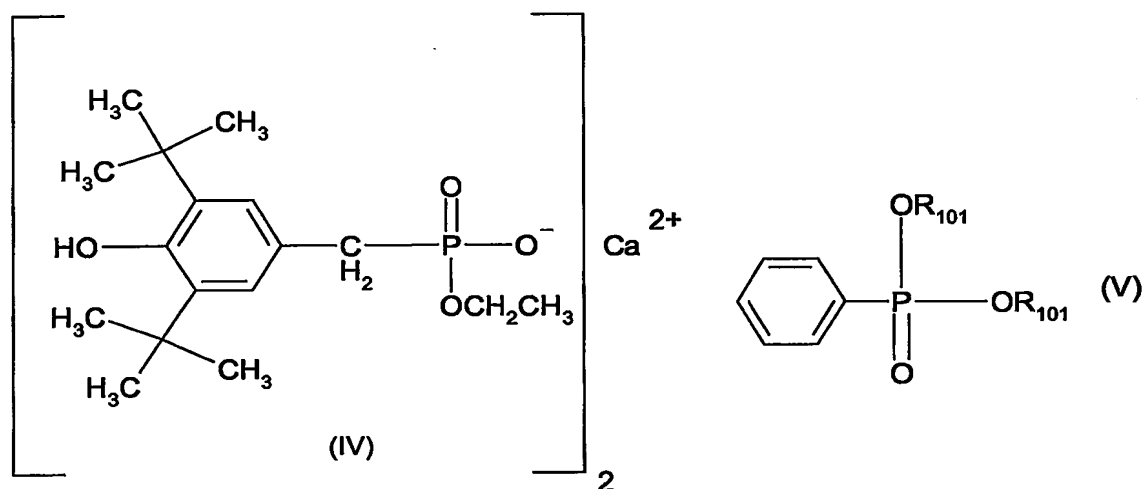
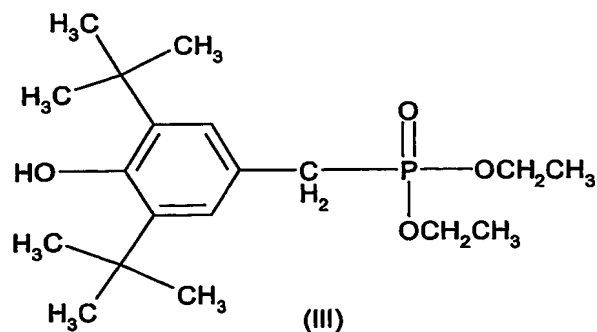
R₁₀₃ is C₁-C₂₀alkyl, unsubstituted or C₁-C₄alkyl-substituted phenyl or naphthyl,

R₁₀₄ is hydrogen, C₁-C₂₀alkyl, unsubstituted or C₁-C₄alkyl-substituted phenyl or naphthyl; or M^{r+} / r;

M^{r+} is an r-valent metal cation, r is 1, 2, 3 or 4; and

30 n is 1, 2, 3, 4, 5 or 6.

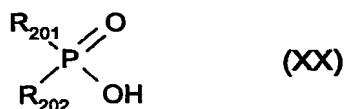
Preferably the phosphonate is of formula III, IV, V, VI or VII



wherein the R_{101} are each independently of one another hydrogen or M^{r+} / r.

10 Some of the compounds of formulae II, IIa, III, IV, V, VI, VII and VIII are commercially available or can be prepared by standard processes, as for example described in US 4 778 840.

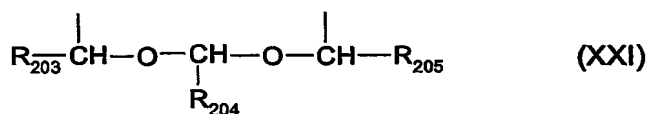
The phosphinates are of the formula XX



wherein

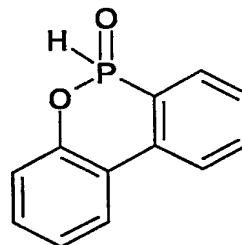
R_{201} is hydrogen, $\text{C}_1\text{-C}_{20}$ alkyl, phenyl or $\text{C}_1\text{-C}_4$ alkyl substituted phenyl; biphenyl, naphthyl, $-\text{CH}_2\text{-O-C}_1\text{-C}_{20}$ alkyl or $-\text{CH}_2\text{-S-C}_1\text{-C}_{20}$ alkyl,

R_{202} is $\text{C}_1\text{-C}_{20}$ alkyl, phenyl or $\text{C}_1\text{-C}_4$ alkyl substituted phenyl; biphenyl, naphthyl, $-\text{CH}_2\text{-O-C}_1\text{-C}_{20}$ alkyl or $-\text{CH}_2\text{-S-C}_1\text{-C}_{20}$ alkyl, or R_1 and R_2 together are a radical of the formula XXI



wherein

R_{203} , R_{204} and R_{205} independently of each other are $\text{C}_1\text{-C}_{20}$ alkyl, phenyl or $\text{C}_1\text{-C}_4$ alkyl substituted phenyl.



A specific phosphinate is for example compound 101

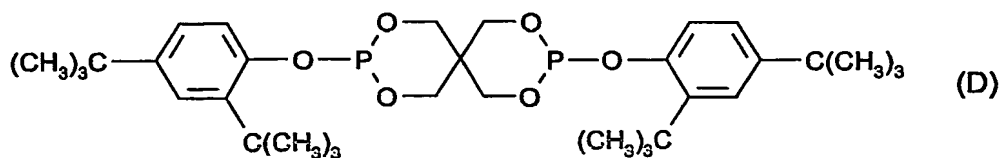
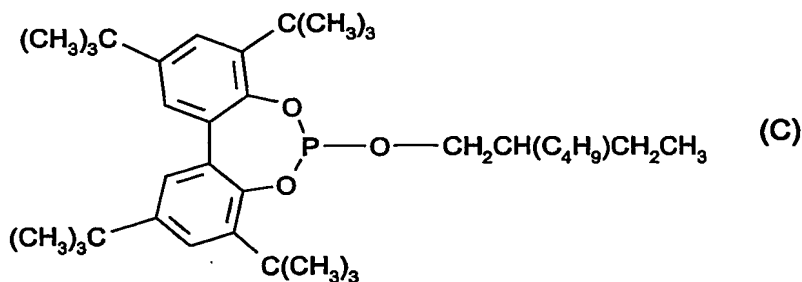
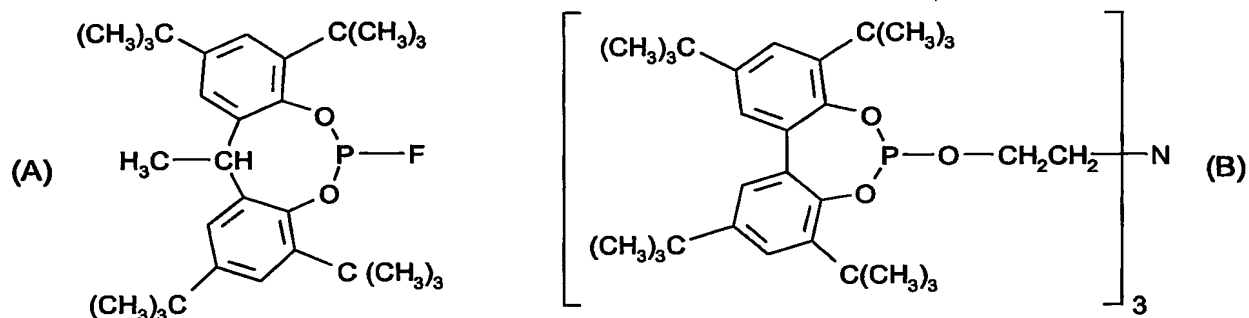
Typical phosphites useful in the instant invention are for example listed below.

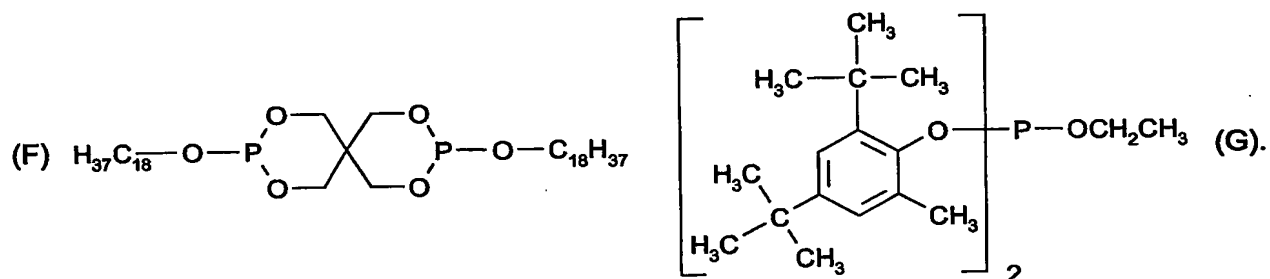
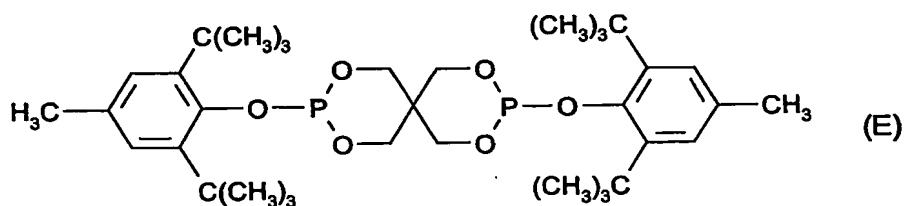
- For example triphenyl phosphite, diphenyl alkyl phosphites, phenyl dialkyl phosphites, tris(nonylphenyl) phosphite, trilauryl phosphite, trioctadecyl phosphite, distearyl pentaerythritol diphosphite, tris(2,4-di-tert-butylphenyl) phosphite, diisodecyl pentaerythritol diphosphite, bis(2,4-di-tert-butylphenyl) pentaerythritol diphosphite, bis(2,6-di-tert-butyl-4-methylphenyl)pentaerythritol diphosphite, diisodecylpentaerythritol diphosphite, bis(2,4-di-tert-butyl-6-methylphenyl)pentaerythritol diphosphite, bis(2,4,6-tris(tert-butyl-

phenyl)pentaerythritol diphosphite, tristearyl sorbitol triphosphite, 6-isooctyloxy-2,4,8,10-tetra-tert-butyl-12H-dibenz[d,g]-1,3,2-dioxaphosphocin, bis(2,4-di-tert-butyl-6-methylphenyl) methyl phosphite, bis(2,4-di-tert-butyl-6-methylphenyl) ethyl phosphite, 6-fluoro-2,4,8,10-tetra-tert-butyl-12-methyl-dibenz[d,g]-1,3,2-dioxaphosphocin, 2,2',2''-nitrido[triethyltris(3,3',5,5'-tetra-tert-butyl-1,1'-biphenyl-2,2'-diyl)phosphite], 2-ethylhexyl(3,3',5,5'-tetra-tert-butyl-1,1'-biphenyl-2,2'-diyl)phosphite, 5-butyl-5-ethyl-2-(2,4,6-tri-tert-butylphenoxy)-1,3,2-dioxaphosphirane.

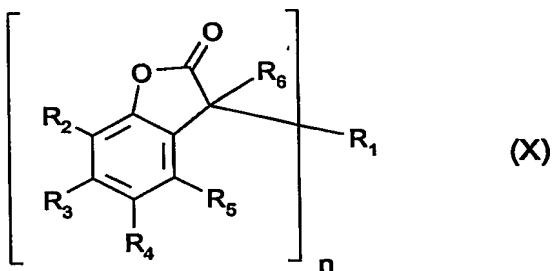
Especially preferred are the following phosphites:

Tris(2,4-di-tert-butylphenyl) phosphite (Irgafos®168, Ciba Specialty Chemicals), tris(nonylphenyl) phosphite,



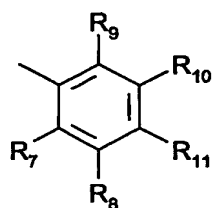


5 For example the benzofuran-2-one type compound is of formula X



wherein, if $n = 1$,

- 10 R_1 is naphthyl, phenanthryl, anthryl, 5,6,7,8-tetrahydro-2-naphthyl, 5,6,7,8-tetrahydro-1-naphthyl, thienyl, benzo[b]thienyl, naphtho[2,3-b]thienyl, thianthrenyl, dibenzofuryl, chromenyl, xanthenyl, phenoxathiinyl, pyrrolyl, imidazolyl, pyrazolyl, pyrazinyl, pyrimidinyl, pyridazinyl, indolizynyl, isoindolyl, indolyl, indazolyl, purinyl, quinolizynyl, isoquinolyl, quinolyl, phthalazinyl, naphthyridinyl, quinoxalinyl, quinazolinyl, cinnolinyl, pteridinyl, carbazolyl, β -carb
- 15 bolinyl, phenanthridinyl, acridinyl, perimidinyl, phenanthrolinyl, phenazinyl, isothiazolyl, phenothiazinyl, isoxazolyl, furazanyl, biphenyl, terphenyl, fluorenyl or phenoxazinyl, each of which is unsubstituted or substituted by C_1 - C_4 alkyl, C_1 - C_4 alkoxy, C_1 - C_4 alkylthio, hydroxy, halogen, amino, C_1 - C_4 alkylamino, phenylamino or di(C_1 - C_4 -alkyl)amino, or R_1 is a radical of formula XI



(XI),

and,

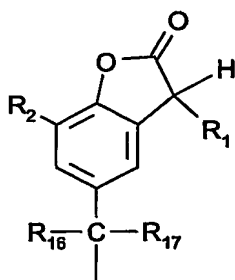
5 if n = 2,

R₁ is unsubstituted or C₁-C₄alkyl- or hydroxy-substituted phenylene or naphthylene; or -R₁₂-X-R₁₃ ,

10 R₂, R₃, R₄ and R₅ are each independently of one another hydrogen, chloro, hydroxy, C₁-C₂₅-alkyl, C₇-C₉phenylalkyl, unsubstituted or C₁-C₄alkyl-substituted phenyl; unsubstituted or C₁-C₄alkyl-substituted C₅-C₈cycloalkyl; C₁-C₁₈alkoxy, C₁-C₁₈alkylthio, C₁-C₄alkylamino, di(C₁-C₄-alkyl)amino, C₁-C₂₅alkanoyloxy, C₁-C₂₅alkanoylamino, C₃-C₂₅alkenoyloxy;

C₃-C₂₅alkanoyloxy which is interrupted by oxygen, sulfur or >N-R_{14} ; C₆-C₈cycloalkylcar-

15 bonyloxy, benzoyloxy or C₁-C₁₂alkyl-substituted benzoyloxy; or R₂ and R₃, or R₃ and R₄, or R₄ and R₅, together with the linking carbon atoms, form a benzene ring, R₄ is additionally -(CH₂)_p-COR₁₅ or -(CH₂)_qOH or, if R₃, R₅ and R₆ are hydrogen, R₄ is additionally a radical of formula XII

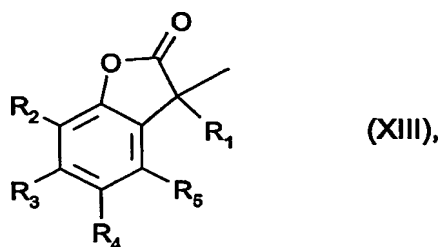


(XII),

20

wherein R₁ is as defined above for n = 1,

R₂ is hydrogen or a radical of formula XIII



wherein R_4 is not a radical of formula XII, and R_1 is as defined above for $n = 1$,

R_7 , R_8 , R_9 , R_{10} and R_{11} are each independently of one another hydrogen, halogen, hydroxy,

5 C_1 - C_{25} alkyl; C_2 - C_{25} alkyl which is interrupted by oxygen, sulfur or $\text{>N}-R_{14}$; C_1 - C_{25} alkoxy;

C_2 - C_{25} alkoxy which is interrupted by oxygen, sulfur or $\text{>N}-R_{14}$; C_1 - C_{25} alkylthio, C_3 - C_{25} -

alkenyl, C_3 - C_{25} alkenyloxy, C_3 - C_{25} alkynyl, C_3 - C_{25} alkynyloxy, C_7 - C_9 phenylalkyl, C_7 - C_9 phenyl-
alkoxy, unsubstituted or C_1 - C_4 alkyl-substituted phenyl; unsubstituted or C_1 - C_4 alkyl-substi-
tuted phenoxy; unsubstituted or C_1 - C_4 alkyl-substituted C_5 - C_8 cycloalkyl; unsubstituted or

10 C_1 - C_4 alkyl-substituted C_5 - C_8 cycloalkoxy; C_1 - C_4 alkylamino, di(C_1 - C_4 alkyl)amino,

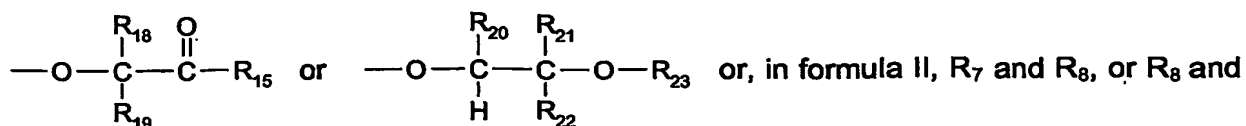
C_1 - C_{25} alkanoyl; C_3 - C_{25} alkanoyl which is interrupted by oxygen, sulfur or $\text{>N}-R_{14}$;

C_1 - C_{25} alkanoyloxy; C_3 - C_{25} alkanoyloxy which is interrupted by oxygen, sulfur or $\text{>N}-R_{14}$;

C_1 - C_{25} alkanoylamino, C_3 - C_{25} alkenoyl; C_3 - C_{25} alkenoyl which is interrupted by oxygen, sulfur
or $\text{>N}-R_{14}$; C_3 - C_{25} alkenoyloxy; C_3 - C_{25} alkenoyloxy which is interrupted by oxygen, sulfur

15 or $\text{>N}-R_{14}$; C_6 - C_9 cycloalkylcarbonyl, C_6 - C_9 cycloalkylcarbonyloxy, benzoyl or

C_1 - C_{12} alkyl-substituted benzoyl; benzoyloxy or C_1 - C_{12} alkyl-substituted benzoyloxy;



R_{11} , together with the linking carbon atoms, form a benzene ring,

R_{12} and R_{13} are each independently of the other unsubstituted or C_1 - C_4 alkyl-substituted phenylene or naphthylene,

R_{14} is hydrogen or C_1 - C_8 alkyl,

R_{15} is hydroxy, $\left[-O^- \frac{1}{r} M^{r+} \right]$, C_1 - C_{18} alkoxy or $-N \begin{matrix} R_{24} \\ R_{25} \end{matrix}$,

- 5 R_{16} and R_{17} are each independently of the other hydrogen, CF_3 , C_1 - C_{12} alkyl or phenyl, or R_{16} and R_{17} , together with the linking carbon atom, are a C_5 - C_8 cycloalkylidene ring which is unsubstituted or substituted by 1 to 3 C_1 - C_4 alkyl;

R_{18} and R_{19} are each independently of the other hydrogen, C_1 - C_4 alkyl or phenyl,

R_{20} is hydrogen or C_1 - C_4 alkyl,

- 10 R_{21} is hydrogen, unsubstituted or C_1 - C_4 alkyl-substituted phenyl; C_1 - C_{25} alkyl; C_2 - C_{25} alkyl

which is interrupted by oxygen, sulfur or $\text{>N}-R_{14}$; C_7 - C_9 phenylalkyl which is unsubstituted or substituted at the phenyl moiety by 1 to 3 C_1 - C_4 alkyl; C_7 - C_{25} phenylalkyl which is

interrupted by oxygen, sulfur or $\text{>N}-R_{14}$ and which is unsubstituted or substituted at the

phenyl moiety by 1 to 3 C_1 - C_4 alkyl, or R_{20} and R_{21} , together with the linking carbon atoms,

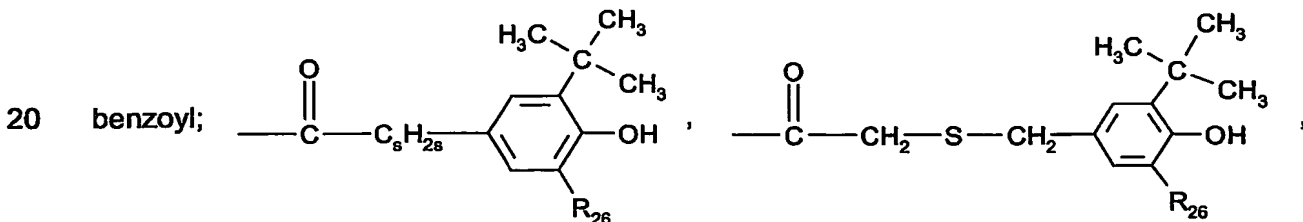
- 15 form a C_5 - C_{12} cycloalkylene ring which is unsubstituted or substituted by 1 to 3 C_1 - C_4 alkyl;

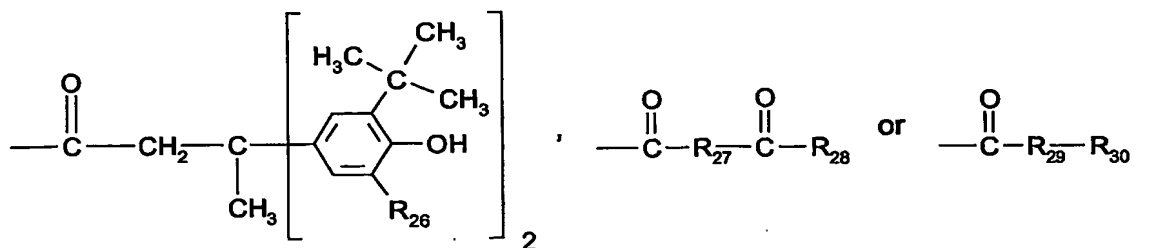
R_{22} is hydrogen or C_1 - C_4 alkyl,

R_{23} is hydrogen, C_1 - C_{25} alkanoyl, C_3 - C_{25} alkenoyl; C_3 - C_{25} alkanoyl which is interrupted by

oxygen, sulfur or $\text{>N}-R_{14}$; C_2 - C_{25} alkanoyl which is substituted by a di(C_1 - C_6 alkyl)phos-

phonate group; C_6 - C_8 cycloalkylcarbonyl, thenoyl, furoyl, benzoyl or C_1 - C_{12} alkyl-substituted



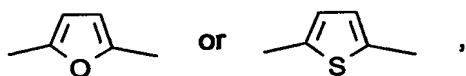


R_{24} and R_{25} are each independently of the other hydrogen or $\text{C}_1\text{-C}_{18}$ alkyl,

R_{26} is hydrogen or $\text{C}_1\text{-C}_8$ alkyl,

R_{27} is a direct bond, $\text{C}_1\text{-C}_{18}$ alkylene; $\text{C}_2\text{-C}_{18}$ alkylene which is interrupted by oxygen, sulfur or

- 5 $\text{N}-\text{R}_{14}$; $\text{C}_2\text{-C}_{18}$ alkenylene, $\text{C}_2\text{-C}_{20}$ alkylidene, $\text{C}_7\text{-C}_{20}$ phenylalkylidene, $\text{C}_5\text{-C}_8$ cycloalkylene, $\text{C}_7\text{-C}_8$ bicycloalkylene, unsubstituted or $\text{C}_1\text{-C}_4$ alkyl-substituted phenylene,



R_{28} is hydroxy, $\left[-\text{O}^- \frac{1}{r} \text{M}^{r+} \right]$, $\text{C}_1\text{-C}_{18}$ alkoxy or $\begin{array}{c} \text{R}_{24} \\ \diagup \\ \text{N} \\ \diagdown \\ \text{R}_{25} \end{array}$,

R_{29} is oxygen, $-\text{NH}-$ or $\begin{array}{c} \text{O} \\ \parallel \\ \text{N}-\text{C}-\text{NH}-\text{R}_{30} \end{array}$,

- 10 R_{30} is $\text{C}_1\text{-C}_{18}$ alkyl or phenyl,

R_{31} is hydrogen or $\text{C}_1\text{-C}_{18}$ alkyl,

M is an r -valent metal cation,

X is a direct bond, oxygen, sulfur or $-\text{NR}_{31}-$,

n is 1 or 2,

- 15 p is 0, 1 or 2,

q is 1, 2, 3, 4, 5 or 6,

r is 1, 2 or 3, and

s is 0, 1 or 2.

- 20 R_1 may be a heterocycle which is naphthyl, phenanthryl, anthryl, 5,6,7,8-tetrahydro-2-naphthyl, 5,6,7,8-tetrahydro-1-naphthyl, thienyl, benzo[b]thienyl, naphtho[2,3-b]thienyl, thianthrenyl, dibenzofuryl, chromenyl, xanthenyl, phenoxathiinyl, pyrrolyl, imidazolyl,

pyrazolyl, pyrazinyl, pyrimidinyl, pyridazinyl, indolizinyl, isoindolyl, indolyl, indazolyl, purinyl, quinolizinyl, isoquinolyl, quinolyl, phthalazinyl, naphthyridinyl, quinoxalinyl, quinazolinyl, cinnolinyl, pteridinyl, carbazolyl, β -carbolinyl, phenanthridinyl, acridinyl, perimidinyl, phenanthrolinyl, phenazinyl, isothiazolyl, phenothiazinyl, isoxazolyl, furazanyl, biphenyl, 5 terphenyl, fluorenyl or phenoxazinyl, each of which is unsubstituted or substituted by C₁-C₄alkyl, C₁-C₄alkoxy, C₁-C₄alkylthio, hydroxy, halogen, amino, C₁-C₄alkylamino, phenylamino or di(C₁-C₄alkyl)amino are, for example, 1-naphthyl, 2-naphthyl, 1-phenylamino-4-naphthyl, 1-methylnaphthyl, 2-methylnaphthyl, 1-methoxy-2-naphthyl, 2-methoxy-1-naphthyl, 1-dimethylamino-2-naphthyl, 1,2-dimethyl-4-naphthyl, 1,2-dimethyl-6-naphthyl, 1,2-dimethyl-7-naphthyl, 10 naphthyl, 1,3-dimethyl-6-naphthyl, 1,4-dimethyl-6-naphthyl, 1,5-dimethyl-2-naphthyl, 1,6-dimethyl-2-naphthyl, 1-hydroxy-2-naphthyl, 2-hydroxy-1-naphthyl, 1,4-dihydroxy-2-naphthyl, 7-phenanthryl, 1-anthryl, 2-anthryl, 9-anthryl, 3-benzo[b]thienyl, 5-benzo[b]thienyl, 2-benzo[b]thienyl, 4-dibenzofuryl, 4,7-dibenzofuryl, 4-methyl-7-dibenzofuryl, 2-xanthenyl, 8-methyl-2-xanthenyl, 3-xanthenyl, 2-phenoxathiinyl, 2,7-phenoxathiinyl, 2-pyrrolyl, 3-pyrrolyl, 15 5-methyl-3-pyrrolyl, 2-imidazolyl, 4-imidazolyl, 5-imidazolyl, 2-methyl-4-imidazolyl, 2-ethyl-4-imidazolyl, 2-ethyl-5-imidazolyl, 3-pyrazolyl, 1-methyl-3-pyrazolyl, 1-propyl-4-pyrazolyl, 2-pyrazinyl, 5,6-dimethyl-2-pyrazinyl, 2-indolizinyl, 2-methyl-3-isoindolyl, 2-methyl-1-isoindolyl, 1-methyl-2-indolyl, 1-methyl-3-indolyl, 1,5-dimethyl-2-indolyl, 1-methyl-3-indazolyl, 2,7-dimethyl-8-purinyl, 2-methoxy-7-methyl-8-purinyl, 2-quinolizinyl, 3-isoquinolyl, 6-isoquinolyl, 20 7-isoquinolyl, isoquinolyl, 3-methoxy-6-isoquinolyl, 2-quinolyl, 6-quinolyl, 7-quinolyl, 2-methoxy-3-quinolyl, 2-methoxy-6-quinolyl, 6-phthalazinyl, 7-phthalazinyl, 1-methoxy-6-phthalazinyl, 1,4-dimethoxy-6-phthalazinyl, 1,8-naphthyridin-2-yl, 2-quinoxalinyl, 6-quinoxalinyl, 2,3-dimethyl-6-quinoxalinyl, 2,3-dimethoxy-6-quinoxalinyl, 2-quinazolinyl, 7-quinazolinyl, 2-dimethylamino-6-quinazolinyl, 3-cinnolinyl, 6-cinnolinyl, 7-cinnolinyl, 3-methoxy-7-cinnolinyl, 25 cinnolinyl, 2-pteridinyl, 6-pteridinyl, 7-pteridinyl, 6,7-dimethoxy-2-pteridinyl, 2-carbazolyl, 3-carbazolyl, 9-methyl-2-carbazolyl, 9-methyl-3-carbazolyl, β -carbolin-3-yl, 1-methyl- β -carbolin-3-yl, 1-methyl- β -carbolin-6-yl, 3-phenanthridinyl, 2-acridinyl, 3-acridinyl, 2-perimidinyl, 1-methyl-5-perimidinyl, 5-phenanthrolinyl, 6-phenanthrolinyl, 1-phenazinyl, 2-phenazinyl, 3-isothiazolyl, 4-isothiazolyl, 5-isothiazolyl, 2-phenothiazinyl, 3-phenothiazinyl, 10-methyl-3-phenothiazinyl, 3-isoxazolyl, 4-isoxazolyl, 5-isoxazolyl, 4-methyl-3-furazanyl, 2-phenoxazinyl 30 or 10-methyl-2-phenoxazinyl.

Particularly preferred are naphthyl, phenanthryl, anthryl, 5,6,7,8-tetrahydro-2-naphthyl, 5,6,7,8-tetrahydro-1-naphthyl, thienyl, benzo[b]thienyl, naphtho[2,2-b]thienyl, phenanthro[2,2-b]thienyl.

dibenzofuryl, chromenyl, xanthenyl, phenoxathiinyl, pyrrolyl, isoindolyl, indolyl, phenothiazinyl, biphenyl, terphenyl, fluorenyl or phenoxazinyl, each of which is unsubstituted or substituted by C₁-C₄alkyl, C₁-C₄alkoxy, C₁-C₄alkylthio, hydroxy, phenylamino or di(C₁-C₄-alkyl)-amino, for example 1-naphthyl, 2-naphthyl, 1-phenylamino-4-naphthyl, 1-methylnaphthyl, 2-methylnaphthyl, 1-methoxy-2-naphthyl, 2-methoxy-1-naphthyl, 1-dimethylamino-2-naphthyl, 1,2-dimethyl-4-naphthyl, 1,2-dimethyl-6-naphthyl, 1,2-dimethyl-7-naphthyl, 1,3-dimethyl-6-naphthyl, 1,4-dimethyl-6-naphthyl, 1,5-dimethyl-2-naphthyl, 1,6-dimethyl-2-naphthyl, 1-hydroxy-2-naphthyl, 2-hydroxy-1-naphthyl, 1,4-dihydroxy-2-naphthyl, 7-phenanthryl, 1-anthryl, 2-anthryl, 9-anthryl, 3-benzo[b]thienyl, 5-benzo[b]thienyl, 2-benzo[b]thienyl, 4-dibenzofuryl, 4,7-dibenzofuryl, 4-methyl-7-dibenzofuryl, 2-xanthenyl, 8-methyl-2-xanthenyl, 3-xanthenyl, 2-pyrrolyl, 3-pyrrolyl, 2-phenothiazinyl, 3-phenothiazinyl, 10-methyl-3-phenothiazinyl.

Halogen is typically chloro, bromo or iodo. Chloro is preferred.

Alkanoyl of up to 25 carbon atoms is a branched or unbranched radical, typically formyl, acetyl, propionyl, butanoyl, pentanoyl, hexanoyl, heptanoyl, octanoyl, nonanoyl, decanoyl, undecanoyl, dodecanoyl, tridecanoyl, tetradecanoyl, pentadecanoyl, hexadecanoyl, heptadecanoyl, octadecanoyl, eicosanoyl or docosanoyl. Alkanoyl of 2 to 18, in particular of 2 to 12, e.g. of 2 to 6, carbon atoms is preferred. Acetyl is particularly preferred.

C₂-C₂₅Alkanoyl which is substituted by a di(C₁-C₆alkyl)phosphonate group is typically (CH₃CH₂O)₂POCH₂CO-, (CH₃O)₂POCH₂CO-, (CH₃CH₂CH₂CH₂O)₂POCH₂CO-, (CH₃CH₂O)₂POCH₂CH₂CO-, (CH₃O)₂POCH₂CH₂CO-, (CH₃CH₂CH₂CH₂O)₂POCH₂CH₂CO-, (CH₃CH₂O)₂PO(CH₂)₄CO-, (CH₃CH₂O)₂PO(CH₂)₈CO- or (CH₃CH₂O)₂PO(CH₂)₁₇CO-.

Alkanoyloxy of up to 25 carbon atoms is a branched or unbranched radical, typically formyloxy, acetoxy, propionyloxy, butanoyloxy, pentanoyloxy, hexanoyloxy, heptanoyloxy, octanoyloxy, nonanoyloxy, decanoyloxy, undecanoyloxy, dodecanoyloxy, tridecanoyloxy, tetradecanoyloxy, pentadecanoyloxy, hexadecanoyloxy, heptadecanoyloxy, octadecanoyloxy, eicosanoyloxy or docosanoyloxy. Alkanoyloxy of 2 to 18, in particular of 2 to 12, e.g. of 2 to 6, carbon atoms is preferred. Acetoxy is particularly preferred.

Alkenoyl of 3 to 25 carbon atoms is a branched or unbranched radical, typically propenoyl, 2-butenoyl, 3-butenoyl, isobutenoyl, n-2,4-pentadienoyl, 3-methyl-2-butenoyl, n-2-octenoyl, n-

2-dodecenoyl, isododecenoyl, oleoyl, n-2-octadecenoyl or n-4-octadecenoyl. Alkenoyl of 3 to 18, preferably of 3 to 12, e.g. of 3 to 6, most preferably of 3 to 4, carbon atoms is preferred.

C₃-C₂₅Alkenoyl which is interrupted by oxygen, sulfur or $\text{N}-\text{R}_{14}$ is typically

5 CH₃OCH₂CH₂CH=CHCO- or CH₃OCH₂CH₂OCH=CHCO-.

Alkenoyloxy of 3 to 25 carbon atoms is a branched or unbranched radical, typically propenoyloxy, 2-butenoyloxy, 3-butenoyloxy, isobutenoyloxy, n-2,4-pentadienoyloxy, 3-methyl-2-butenoyloxy, n-2-octenoyloxy, n-2-dodecenoyloxy, isododecenoyloxy, oleoyloxy, n-2-octadecenoyloxy or n-4-octadecenoyloxy. Alkenoyloxy of 3 to 18, preferably of 3 to 12, e.g. of 3 to 6, most preferably of 3 to 4, carbon atoms is preferred.

C₃-C₂₅Alkenoyloxy which is interrupted by oxygen, sulfur or $\text{N}-\text{R}_{14}$ is typically

15 CH₃OCH₂CH₂CH=CHCOO- or CH₃OCH₂CH₂OCH=CHCOO-.

C₃-C₂₅Alkanoyl which is interrupted by oxygen, sulfur or $\text{N}-\text{R}_{14}$ is typically

CH₃-O-CH₂CO-, CH₃-S-CH₂CO-, CH₃-NH-CH₂CO-, CH₃-N(CH₃)-CH₂CO-,
CH₃-O-CH₂CH₂-O-CH₂CO-, CH₃-(O-CH₂CH₂)₂O-CH₂CO-, CH₃-(O-CH₂CH₂)₃O-CH₂CO- or
CH₃-(O-CH₂CH₂)₄O-CH₂CO-.

20

C₃-C₂₅Alkanoyloxy which is interrupted by oxygen, sulfur or $\text{N}-\text{R}_{14}$ is typically

CH₃-O-CH₂COO-, CH₃-S-CH₂COO-, CH₃-NH-CH₂COO-, CH₃-N(CH₃)-CH₂COO-,
CH₃-O-CH₂CH₂-O-CH₂COO-, CH₃-(O-CH₂CH₂)₂O-CH₂COO-,
CH₃-(O-CH₂CH₂)₃O-CH₂COO- or CH₃-(O-CH₂CH₂)₄O-CH₂COO-.

25

C₆-C₉Cycloalkylcarbonyl is typically cyclohexylcarbonyl, cycloheptylcarbonyl or cyclooctylcarbonyl. Cyclohexylcarbonyl is preferred.

C₆-C₉Cycloalkylcarbonyloxy is typically cyclohexylcarbonyloxy, cycloheptylcarbonyloxy or cyclooctylcarbonyloxy. Cyclohexylcarbonyloxy is preferred.

5 C₁-C₁₂Alkyl-substituted benzoyl which preferably carries 1 to 3, more preferably 1 or 2, alkyl groups is typically o-, m- or p-methylbenzoyl, 2,3-dimethylbenzoyl, 2,4-dimethylbenzoyl, 2,5-dimethylbenzoyl, 2,6-dimethylbenzoyl, 3,4-dimethylbenzoyl, 3,5-dimethylbenzoyl, 2-methyl-6-ethylbenzoyl, 4-tert-butylbenzoyl, 2-ethylbenzoyl, 2,4,6-trimethylbenzoyl, 2,6-dimethyl-4-tert-butylbenzoyl or 3,5-di-tert-butylbenzoyl. Preferred substituents are C₁-C₈alkyl, in particular C₁-C₄alkyl.

10 C₁-C₁₂Alkyl-substituted benzoyloxy which preferably carries 1 to 3, more preferably 1 or 2, alkyl groups is typically o-, m- or p-methylbenzoyloxy, 2,3-dimethylbenzoyloxy, 2,4-dimethylbenzoyloxy, 2,5-dimethylbenzoyloxy, 2,6-dimethylbenzoyloxy, 3,4-dimethylbenzoyloxy, 3,5-dimethylbenzoyloxy, 2-methyl-6-ethylbenzoyloxy, 4-tert-butylbenzoyloxy, 2-ethylbenzoyloxy, 15 2,4,6-trimethylbenzoyloxy, 2,6-dimethyl-4-tert-butylbenzoyloxy or 3,5-di-tert-butylbenzoyloxy. Preferred substituents are C₁-C₈alkyl, in particular C₁-C₄alkyl.

20 Alkyl of up to 25 carbon atoms is a branched or unbranched radical, such as methyl, ethyl, propyl, isopropyl, n-butyl, sec-butyl, isobutyl, tert-butyl, 2-ethylbutyl, n-pentyl, isopentyl, 1-methylpentyl, 1,3-dimethylbutyl, n-hexyl, 1-methylhexyl, n-heptyl, isoheptyl, 1,1,3,3-tetramethylbutyl, 1-methylheptyl, 3-methylheptyl, n-octyl, 2-ethylhexyl, 1,1,3-trimethylhexyl, 1,1,3,3-tetramethylpentyl, nonyl, decyl, undecyl, 1-methylundecyl, dodecyl, 1,1,3,3,5,5-hexamethylhexyl, tridecyl, tetradecyl, pentadecyl, hexadecyl, heptadecyl, octadecyl, eicosyl or docosyl. One of the preferred meanings of R₂ and R₄ is, for example, C₁-C₁₈alkyl. A particularly 25 preferred meaning of R₄ is C₁-C₄alkyl.

30 Alkenyl of 3 to 25 carbon atoms is a branched or unbranched radical, such as propenyl, 2-butenyl, 3-butenyl, isobutenyl, n-2,4-pentadienyl, 3-methyl-2-butenyl, n-2-octenyl, n-2-dodecenyl, isododecenyl, oleyl, n-2-octadecenyl or n-4-octadecenyl. Alkenyl of 3 to 18, preferably of 3 to 12, e.g. of 3 to 6, in particular of 3 to 4, carbon atoms is preferred.

Alkenyloxy of 3 to 25 carbon atoms is a branched or unbranched radical, such as propenyloxy, 2-butenyloxy, 3-butenyloxy, isobutenyloxy, n-2,4-pentadienyloxy, 3-methyl-2-butenyloxy, n-2-octenyloxy, n-2-dodecenyloxy, isododecenyloxy, oleyloxy, n-2-octadecenyloxy or n-4-

octadecenyloxy. Alkenyloxy of 3 to 18, preferably of 3 to 12, e.g. of 3 to 6, in particular of 3 to 4, carbon atoms is preferred.

Alkynyl of 3 to 25 carbon atoms is a branched or unbranched radical, such as propynyl
5 ($-\text{CH}_2-\text{C}\equiv\text{CH}$), 2-butylnyl, 3-butylnyl, n-2-octynyl, or n-2-dodecynyl. Alkynyl of 3 to 18, preferably of 3 to 12, e.g. of 3 to 6, in particular of 3 to 4 carbon atoms is preferred.

Alkynyloxy of 3 to 25 carbon atoms is a branched or unbranched radical, such propynyloxy
($-\text{OCH}_2-\text{C}\equiv\text{CH}$) 2-butynyloxy, 3-butynyloxy, n-2-octynyloxy, or n-2-dodecynyloxy.
10 Alkynyloxy of 3 to 18, preferably of 3 to 12, e.g. of 3 to 6, in particular of 3 to 4, carbon atoms is preferred.

$\text{C}_2\text{-C}_{25}$ Alkyl which is interrupted by oxygen, sulfur or $\text{N}-\text{R}_{14}$ is typically

$\text{CH}_3\text{-O-CH}_2\text{-}$, $\text{CH}_3\text{-S-CH}_2\text{-}$, $\text{CH}_3\text{-NH-CH}_2\text{-}$, $\text{CH}_3\text{-N(CH}_3\text{)-CH}_2\text{-}$, $\text{CH}_3\text{-O-CH}_2\text{CH}_2\text{-O-CH}_2\text{-}$,
15 $\text{CH}_3\text{-(O-CH}_2\text{CH}_2\text{)}_2\text{-O-CH}_2\text{-}$, $\text{CH}_3\text{-(O-CH}_2\text{CH}_2\text{)}_3\text{-O-CH}_2\text{-}$ or $\text{CH}_3\text{-(O-CH}_2\text{CH}_2\text{)}_4\text{-O-CH}_2\text{-}$.

$\text{C}_7\text{-C}_9$ Phenylalkyl is typically benzyl, α -methylbenzyl, α,α -dimethylbenzyl or 2-phenylethyl. Benzyl and α,α -dimethylbenzyl are preferred.

20 $\text{C}_7\text{-C}_9$ Phenylalkyl which is unsubstituted or substituted at the phenyl moiety by 1 to 3 $\text{C}_1\text{-C}_4$ -alkyl is typically benzyl, α -methylbenzyl, α,α -dimethylbenzyl, 2-phenylethyl, 2-methylbenzyl, 3-methylbenzyl, 4-methylbenzyl, 2,4-dimethylbenzyl, 2,6-dimethylbenzyl or 4-tert-butylbenzyl. Benzyl is preferred.

25 $\text{C}_7\text{-C}_{25}$ Phenylalkyl which is interrupted by oxygen, sulfur or $\text{N}-\text{R}_{14}$ and which is unsubstituted or substituted at the phenyl moiety by 1 to 3 $\text{C}_1\text{-C}_4$ alkyl is a branched or unbranched radical, such as phenoxymethyl, 2-methylphenoxymethyl, 3-methyl-phenoxymethyl, 4-methylphenoxymethyl, 2,4-dimethylphenoxymethyl, 2,3-dimethylphenoxymethyl, phenylthiomethyl, N-methyl-N-phenyl-methyl, N-ethyl-N-phenylmethyl, 4-tert-butylphenoxymethyl, 4-tert-butylphenylthiomethyl, 2,4-di-tert-butylphenoxymethyl, 2,6-di-tert-butylphenoxymethyl,

methyl, phenoxyethoxyethoxyethoxymethyl, benzyloxymethyl, benzyloxyethoxymethyl, N-benzyl-N-ethylmethyl or N-benzyl-N-isopropylmethyl.

5 C₇-C₉Phenylalkoxy is typically benzyloxy, α-methylbenzyloxy, α,α-dimethylbenzyloxy or 2-phenylethoxy. Benzyloxy is preferred.

10 C₁-C₄Alkyl-substituted phenyl which preferably contains 1 to 3, in particular 1 or 2, alkyl groups is typically o-, m- or p-methylphenyl, 2,3-dimethylphenyl, 2,4-dimethylphenyl, 2,5-dimethylphenyl, 2,6-dimethylphenyl, 3,4-dimethylphenyl, 3,5-dimethylphenyl, 2-methyl-6-ethylphenyl, 4-tert-butylphenyl, 2-ethylphenyl or 2,6-diethylphenyl.

15 C₁-C₄Alkyl-substituted phenoxy which preferably contains 1 to 3, in particular 1 or 2, alkyl groups is typically o-, m- or p-methylphenoxy, 2,3-dimethylphenoxy, 2,4-dimethylphenoxy, 2,5-dimethylphenoxy, 2,6-dimethylphenoxy, 3,4-dimethylphenoxy, 3,5-dimethylphenoxy, 2-methyl-6-ethylphenoxy, 4-tert-butylphenoxy, 2-ethylphenoxy or 2,6-diethylphenoxy.

20 Unsubstituted or C₁-C₄alkyl-substituted C₅-C₈cycloalkyl is, for example, cyclopentyl, methylcyclopentyl, dimethylcyclopentyl, cyclohexyl, methylcyclohexyl, dimethylcyclohexyl, trimethylcyclohexyl, tert-butylcyclohexyl, cycloheptyl or cyclooctyl. Cyclohexyl and tert-butylcyclohexyl are preferred.

25 Unsubstituted or C₁-C₄alkyl-substituted C₅-C₈cycloalkoxy is, for example, cyclopentoxo, methylcyclopentoxo, dimethylcyclopentoxo, cyclohexoxo, methylcyclohexoxo, dimethylcyclohexoxo, trimethylcyclohexoxo, tert-butylcyclohexoxo, cycloheptoxo or cyclooctoxo. Cyclohexoxo and tert-butylcyclohexoxo are preferred.

30 Alkoxy of up to 25 carbon atoms is a branched or unbranched radical, such as methoxy, ethoxy, propoxy, isopropoxy, n-butoxy, isobutoxy, pentoxy, isopentoxy, hexoxy, heptoxy, octoxy, decyloxy, tetradecyloxy, hexadecyloxy or octadecyloxy. Alkoxy of 1 to 12, in particular of 1 to 8, e.g. of 1 to 6, carbon atoms is preferred.

C₂-C₂₅Alkoxy which is interrupted by oxygen, sulfur or >N-R_{14} is typically

CH₃-O-CH₂CH₂O-, CH₃-S-CH₂CH₂O-, CH₃-NH-CH₂CH₂O-, CH₃-N(CH₃)-CH₂CH₂O-,

$\text{CH}_3\text{-O-CH}_2\text{CH}_2\text{-O-CH}_2\text{CH}_2\text{O-}$, $\text{CH}_3\text{-(O-CH}_2\text{CH}_2\text{)}_2\text{O-CH}_2\text{CH}_2\text{O-}$,
 $\text{CH}_3\text{-(O-CH}_2\text{CH}_2\text{)}_3\text{O-CH}_2\text{CH}_2\text{O-}$ or $\text{CH}_3\text{-(O-CH}_2\text{CH}_2\text{)}_4\text{O-CH}_2\text{CH}_2\text{O-}$.

5 Alkylthio of up to 25 carbon atoms is a branched or unbranched radical, such as methylthio, ethylthio, propylthio, isopropylthio, n-butylthio, isobutylthio, pentylthio, isopentylthio, hexylthio, heptylthio, octylthio, decylthio, tetradecylthio, hexadecylthio or octadecylthio. Alkylthio of 1 to 12, in particular of 1 to 8, e.g. of 1 to 6 carbon atoms is preferred.

10 Alkylamino of up to 4 carbon atoms is a branched or unbranched radical, such as methylamino, ethylamino, propylamino, isopropylamino, n-butylamino, isobutylamino or tert-butylamino.

15 Di(C₁-C₄alkyl)amino also means that the two radicals are independently of the other branched or unbranched, such as dimethylamino, methylethylamino, diethylamino, methyl-n-propylamino, methylisopropylamino, methyl-n-butylamino, methylisobutylamino, ethylisopropylamino, ethyl-n-butylamino, ethylisobutylamino, ethyl-tert-butylamino, diethylamino, diisopropylamino, isopropyl-n-butylamino, isopropylisobutylamino, di-n-butylamino or di-isobutylamino.

20 Alkanoylamino of up to 25 carbon atoms is a branched or unbranched radical, such as formylamino, acetylamino, propionylamino, butanoylamino, pentanoylamino, hexanoylamino, heptanoylamino, octanoylamino, nonanoylamino, decanoylamino, undecanoylamino, dodecanoylamino, tridecanoylamino, tetradecanoylamino, pentadecanoylamino, hexadecanoylamino, heptadecanoylamino, octadecanoylamino, eicosanoylamino or docosanoylamino.

25 Alkanoylamino of 2 to 18, in particular of 2 to 12, e.g. of 2 to 6, carbon atoms is preferred.

30 C₁-C₁₈Alkylene is a branched or unbranched radical, such as methylene, ethylene, propylene, trimethylene, tetramethylene, pentamethylene, hexamethylene, heptamethylene, octamethylene, decamethylene, dodecamethylene or octadecamethylene. C₁-C₁₂Alkylene and, in particular, C₁-C₈alkylene are preferred.

A C₁-C₄Alkyl-substituted C₅-C₁₂cycloalkylene ring which preferably contains 1 to 3, in particular 1 or 2, branched or unbranched alkyl groups is typically cyclopentylene, methylcyclopentylene, dimethylcyclopentylene, cyclohexylene, methylcyclohexylene, dimethylcyclohexylene, cycloheptylene, methylcycloheptylene, dimethylcycloheptylene, cyclooctylene, methylcyclooctylene, dimethylcyclooctylene, cyclononylene, methylcyclononylene, dimethylcyclononylene, cyclodecylene, methylcyclodecylene, dimethylcyclodecylene, cycloundecylene, methylcycloundecylene, dimethylcycloundecylene, cyclododecylene, methylcyclododecylene, dimethylcyclododecylene.

lene, trimethylcyclohexylene, tert-butylcyclohexylene, cycloheptylene, cyclooctylene or cyclo-decylene. Cyclohexylene and tert-butylcyclohexylene are preferred.

C₂-C₁₈Alkylene which is interrupted by oxygen, sulfur or >N-R_{14} is, for example,

- 5 -CH₂-O-CH₂-, -CH₂-S-CH₂-, -CH₂-NH-CH₂-, -CH₂-N(CH₃)-CH₂-, -CH₂-O-CH₂CH₂-O-CH₂-,
-CH₂-(O-CH₂CH₂)₂-O-CH₂-, -CH₂-(O-CH₂CH₂)₃-O-CH₂-, -CH₂-(O-CH₂CH₂)₄-O-CH₂- or
-CH₂CH₂-S-CH₂CH₂-.

- 10 C₂-C₁₈Alkenylene is typically vinylene, methylvinylene, octenylethylene or dodecenylethy-
lene. C₂-C₈Alkenylene is preferred.

Alkylidene of 2 to 20 carbon atoms is, for example, ethylidene, propylidene, butylidene, pen-
tylidene, 4-methylpentylidene, heptylidene, nonylidene, tridecylidene, nonadecylidene, 1-me-
thylethylidene, 1-ethylpropylidene or 1-ethylpentylidene. C₂-C₈Alkylidene is preferred.

- 15 Phenylalkylidene of 7 to 20 carbon atoms is typically benzylidene, 2-phenylethylidene or 1-
phenyl-2-hexylidene. C₇-C₉Phenylalkylidene is preferred.

- 20 C₅-C₈Cycloalkylene is a saturated hydrocarbon group having two free valencies and at least
one ring unit and is typically cyclopentylene, cyclohexylene, cycloheptylene or cyclooctylene.
Cyclohexylene is preferred.

C₇-C₈Bicycloalkylene is typically bicycloheptylene or bicyclooctylene.

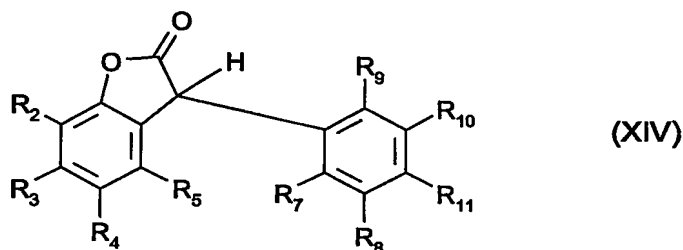
- 25 Unsubstituted or C₁-C₄alkyl-substituted phenylene or naphthylene is typically 1,2-, 1,3-, 1,4-
phenylene, 1,2-, 1,3-, 1,4-, 1,6-, 1,7-, 2,6- or 2,7-naphthylene. 1,4-Phenylene is preferred.

- 30 A C₁-C₄Alkyl-substituted C₅-C₈cycloalkylidene ring which preferably contains 1 to 3, in parti-
cular 1 or 2, branched or unbranched alkyl groups is typically cyclopentylidene, methylcyclo-
pentylidene, dimethylcyclopentylidene, cyclohexylidene, methylcyclohexylidene, dimethylcyc-
lohexylidene, trimethylcyclohexylidene, tert-butylcyclohexylidene, cycloheptylidene or cyclo-
octylidene. Cyclohexylidene and tert-butylcyclohexylidene are preferred.

A mono-, di- or tri-valent metal cation is preferably an alkali metal cation, alkaline earth metal cation or aluminium cation, typically Na^+ , K^+ , Mg^{++} , Ca^{++} or Al^{+++} .

Preferably the benzofuran-2-one type compound is of formula XIV

5



wherein

R_2 is hydrogen or $\text{C}_1\text{-C}_6$ alkyl,

10 R_3 is hydrogen,

R_4 is hydrogen or $\text{C}_1\text{-C}_6$ alkyl,

R_5 is hydrogen,

R_7 , R_8 , R_9 , R_{10} and R_{11} are each independently of one another hydrogen, $\text{C}_1\text{-C}_4$ alkyl, $\text{C}_1\text{-C}_4$ -

alkoxy or $\text{—O—}\begin{array}{c} \text{R}_{20} \quad \text{R}_{21} \\ | \quad | \\ \text{C} \text{—} \text{C} \text{—} \text{O—R}_{23} \\ | \quad | \\ \text{H} \quad \text{R}_{22} \end{array}$, with the proviso that at least two of R_7 , R_8 , R_9 , R_{10} or

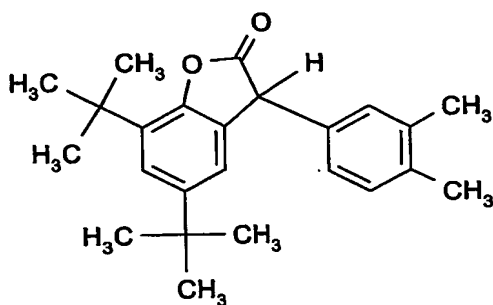
15 R_{11} are hydrogen,

R_{20} , R_{21} and R_{23} are hydrogen, and

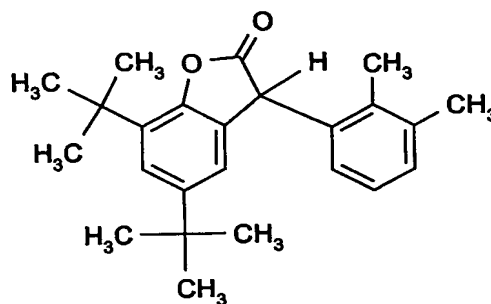
R_{23} is $\text{C}_2\text{-C}_4$ alkanoyl.

In particular the benzofuran-2-one type compound is of formula XIVa or XIVb

20



(XIVa)



(XIVb)

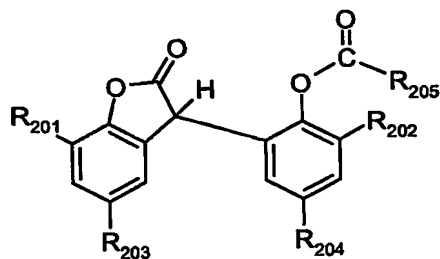
or a mixture or blend of the two compounds of formulae XIVa and XIVb.

The benzofuran-2-one type compounds are known in the literature and partially items of commerce. Their preparation is described, inter alia, in the following U.S. patents: U.S. 4,325,863; U.S. 4,388,244; U.S. 5,175,312; U.S. 5,252,643; U.S. 5,216,052; U.S. 5,369,159; U.S. 5,488,117; U.S. 5,356,966; U.S. 5,367,008; U.S. 5,428,162; U.S. 5,428,177 or U.S. 5,516,920.

Specific commercial examples for a phosphinate is Sanko HCA from Sankyo for phosphonates Irgamod 195 and Irgafos 12 and for phosphites Irgafos 168 from Ciba Specialty Chemicals.

A specific commercial example of a benzofuran-2-one is Irganox HP 136 from Ciba Specialty Chemicals.

Alternatively the benzofuran-2-one type compound is of formula XV



(XV)

wherein

R₂₀₁ and R₂₀₂ are each independently of one another hydrogen or C₁-C₈alkyl, R₂₀₃ and R₂₀₄ are each independently of one another C₁-C₁₂alkyl, and

R₂₀₅ is C₁-C₇alkyl.

Of special interest is the compound of the formula XV wherein

R₂₀₁ and R₂₀₂ are hydrogen,

5 R₂₀₃ and R₂₀₄ are tert-octyl, and

R₂₀₅ is methyl.

The synthesis of the compounds of the formula (XV) is, for example, disclosed in EP-A-0 871 066.

10

For example the bis-acyllactam is used in an amount of 0.01 to 5 %, preferably from 0.1 to 2% by weight based on the weight of the polycondensate.

15

For instance the phosphite, phosphinate or phosphonate is used in an amount of 0.01 to 5 %, preferably 0.01 to 1% by weight based on the weight of the polycondensate.

Typically the benzofuran-2-one type compound is used in an amount of 0.01 to 5 %, preferably 0.01to 1% by weight based on the weight of the polycondensate.

20

For example the sum of the components bis-acyllactam, phosphite, phosphinate or phosphonate and benzofurna-2-one is from 0.2 to 10%, preferably from 0.5 to 3% by weight based on the weight of the polycondensate.

25

The ratio of the bis-acyllactam to the phosphite, phosphinate or phosphonate or the benzofuran-2-one type compound or the sum of all is typically from 1:10 to 5:1.

For example the process is carried out in such a way that the maximum mass-temperature of the melt is from 170° to 320° C.

30

Processing the polycondensate in the melt means heating above the melting point or glass transition temperature usually carried out, with stirring, until the blend is homogeneous. The temperature depends in this case on the polycondensate used. For example:

- 260 to 290°C for fibre- and film-grade PET
- 270 to 310°C for bottle- and industrial-yarn-grade PET

- 240 to 290°C for PBT
- 170 to 240°C for amorphous PET-grades
- 220 to 280°C for thermoplastic elastomers based on polyesters
- 280 to 320°C for PC
- 5 • 270 to 290°C for PA 6.6
- 240 to 270°C for PA 6.

The appropriate processing temperature in dependence on type and grade of polycondensates can be found for example in "Kunststoff Taschenbuch" 21th edition, edited by H. Saechtling, Carl Hanser Verlag 1979.

10

The incorporation can be carried out in any heatable container equipped with a stirrer, e.g. in a closed apparatus such as a kneader, mixer or stirred vessel. The incorporation is preferably carried out in an extruder or in a kneader. It is immaterial whether processing takes place in an inert atmosphere or in the presence of oxygen.

- 15 The addition of the additive or additive blend to the polycondensate can be carried out in all customary mixing machines in which the polycondensate is melted and mixed with the additives. Suitable machines are known to those skilled in the art. They are predominantly mixers, kneaders and extruders.

- 20 The process is preferably carried out in an extruder by introducing the additive during processing.

Particularly preferred processing machines are single-screw extruders, contrarotating and corotating twin-screw extruders, planetary-gear extruders, ring extruders or cokneaders. It is also possible to use processing machines provided with at least one gas removal compartment to which a vacuum can be applied.

- 25 Suitable extruders and kneaders are described, for example, in *Handbuch der Kunststoffextrusion, Vol. 1 Grundlagen*, Editors F. Hensen, W. Knappe, H. Potente, 1989, pp. 3-7, ISBN:3-446-14339-4 (Vol. 2 *Extrusionsanlagen* 1986, ISBN 3-446-14329-7).

- 30 For example, the screw length is 1 - 60 screw diameters, preferably 35-48 screw diameters. The rotational speed of the screw is preferably 10 - 600 rotations per minute (rpm), very particularly preferably 25 - 300 rpm.

If a plurality of components are added, these can be premixed or added individually.

The additives of the invention and optional further additives can also be added to the polycondensate in the form of a masterbatch ("concentrate") which contains the components in a concentration of, for example, about 1 % to about 40% and preferably 2 % to about 20 % by weight incorporated in a polycondensate. The polycondensate must not be necessarily of identical structure than the polycondensate where the additives are added finally. In such operations, the polycondensate can be used in the form of powder, granules, solutions, suspensions or in the form of latices.

Incorporation can take place prior to or during the shaping operation, or by applying the dissolved or dispersed compound to the polycondensate, with or without subsequent evaporation of the solvent.

The processing apparatus is preferably a single-screw extruder, twin-screw extruder, planetary-gear extruder, ring extruder or Ko-kneader having optionally one vent zone to which underpressure is applied.

A preferred process is that, which comprises applying an underpressure of less than 250 mbar, particularly preferably of less than 100 mbar and, very particularly preferably, of less than 50 mbar, to the vent zone.

Another preferred process is that, wherein the processing apparatus is a closely intermeshing twin-screw extruder or ring extruder with screws rotating in the same direction and with a feed section, a transition section, at least one vent zone and a metering zone, the vent zone being separated from the transition section or from another vent zone by a fusible plug.

This separation via a fusible plug can be effected, for example, by a combination of a kneading element and a return screw element.

The processing apparatus preferably has 1-4 vent zones, particularly preferably 1-3.

The process is usually carried out under normal pressure. Typical processing times are from 10 seconds to 10 minutes.

This invention also relates to a composition comprising

a) a polycondensate-

- b) at least one bis-acyllactam;
c1) at least one phosphite, phosphinate or phosphonate; or
c2) at least one benzofuran-2-one type compound or
c3) at least one phosphite, phosphinate or phosphonate and one benzofuran-2-one type compound

Further aspects of the invention are a polycondensate obtainable by a process as described above and the use of a mixture of

a) at least one bis-acyllactam;

b1) at least one phosphite, phosphinate or phosphonate; or

b2) at least one benzofuran-2-one type compound or

b3) at least one phosphite, phosphinate or phosphonate and one benzofuran-2-one type compound

for increasing the molecular weight and/or for the modification of a polycondensate.

The definitions and preferences given above for the process apply also for the other aspects of the invention.

Further additives may be present in the polycondensate in addition to the above mentioned novel additive blend. Examples thereof are listed below.

1. Antioxidants

1.1. Alkylated monophenols, for example 2,6-di-tert-butyl-4-methylphenol, 2-tert-butyl-4,6-dimethylphenol, 2,6-di-tert-butyl-4-ethylphenol, 2,6-di-tert-butyl-4-n-butylphenol, 2,6-di-tert-butyl-4-isobutylphenol, 2,6-dicyclopentyl-4-methylphenol, 2-(α -methylcyclohexyl)-4,6-dimethylphenol, 2,6-dioctadecyl-4-methylphenol, 2,4,6-tricyclohexylphenol, 2,6-di-tert-butyl-4-methoxymethylphenol, nonylphenols which are linear or branched in the side chains, for example 2,6-di-nonyl-4-methylphenol, 2,4-dimethyl-6-(1'-methylundec-1'-yl)phenol, 2,4-dimethyl-6-(1'-methylheptadec-1'-yl)phenol, 2,4-dimethyl-6-(1'-methyltridec-1'-yl)phenol and mixtures thereof.

1.2. Alkylthiomethylphenols, for example 2,4-dioctylthiomethyl-6-tert-butylphenol, 2,4-dioctylthiomethyl-6-methylphenol, 2,4-dioctylthiomethyl-6-ethylphenol, 2,6-di-dodecylthiomethyl-4-nonylphenol.

1.3. Hydroquinones and alkylated hydroquinones, for example 2,6-di-tert-butyl-4-methoxyphenol, 2,5-di-tert-butylhydroquinone, 2,5-di-tert-amylhydroquinone, 2,6-diphenyl-4-octadecyloxyphenol, 2,6-di-tert-butylhydroquinone, 2,5-di-tert-butyl-4-hydroxyanisole, 3,5-di-tert-butyl-4-hydroxyanisole, 3,5-di-tert-butyl-4-hydroxyphenyl stearate, bis-(3,5-di-tert-butyl-4-hydroxyphenyl) adipate.

1.4. Tocopherols, for example α -tocopherol, β -tocopherol, γ -tocopherol, δ -tocopherol and mixtures thereof (Vitamin E).

1.5. Hydroxylated thiodiphenyl ethers, for example 2,2'-thiobis(6-tert-butyl-4-methylphenol), 2,2'-thiobis(4-octylphenol), 4,4'-thiobis(6-tert-butyl-3-methylphenol), 4,4'-thiobis(6-tert-butyl-2-methylphenol), 4,4'-thiobis-(3,6-di-sec-amylphenol), 4,4'-bis(2,6-dimethyl-4-hydroxyphenyl)disulfide.

1.6. Alkylidenebisphenols, for example 2,2'-methylenebis(6-tert-butyl-4-methylphenol), 2,2'-methylenebis(6-tert-butyl-4-ethylphenol), 2,2'-methylenebis[4-methyl-6-(α -methylcyclohexyl)phenol], 2,2'-methylenebis(4-methyl-6-cyclohexylphenol), 2,2'-methylenebis(6-nonyl-4-methylphenol), 2,2'-methylenebis(4,6-di-tert-butylphenol), 2,2'-ethylidenebis(4,6-di-tert-butylphenol), 2,2'-ethylidenebis(6-tert-butyl-4-isobutylphenol), 2,2'-methylenebis[6-(α -methylbenzyl)-4-nonylphenol], 2,2'-methylenebis[6-(α,α -dimethylbenzyl)-4-nonylphenol], 4,4'-methylenebis(2,6-di-tert-butylphenol), 4,4'-methylenebis(6-tert-butyl-2-methylphenol), 1,1-bis(5-tert-butyl-4-hydroxy-2-methylphenyl)butane, 2,6-bis(3-tert-butyl-5-methyl-2-hydroxybenzyl)-4-methylphenol, 1,1,3-tris(5-tert-butyl-4-hydroxy-2-methylphenyl)butane, 1,1-bis(5-tert-butyl-4-hydroxy-2-methylphenyl)-3-n-dodecylmercaptobutane, ethylene glycol bis[3,3-bis(3'-tert-butyl-4'-hydroxyphenyl)butyrate], bis(3-tert-butyl-4-hydroxy-5-methylphenyl)dicyclopentadiene, bis[2-(3'-tert-butyl-2'-hydroxy-5'-methylbenzyl)-6-tert-butyl-4-methylphenyl]terephthalate, 1,1-bis(3,5-dimethyl-2-hydroxyphenyl)butane, 2,2-bis-(3,5-di-tert-butyl-4-hydroxyphenyl)propane, 2,2-bis(5-tert-butyl-4-hydroxy-2-methylphenyl)-4-n-dodecylmercaptobutane, 1,1,5,5-tetra-(5-tert-butyl-4-hydroxy-2-methylphenyl)pentane.

1.7. O-, N- and S-benzyl compounds, for example 3,5,3',5'-tetra-tert-butyl-4,4'-dihydroxydibenzyl ether, octadecyl-4-hydroxy-3,5-dimethylbenzylmercaptoacetate, tridecyl-4-hydroxy-3,5-di-tert-butylbenzylmercaptoacetate, 1-tert-butyl-5-di-tert-butyl-4-hydroxybenzylamine, bis(4-

tert-butyl-3-hydroxy-2,6-dimethylbenzyl)dithioterephthalate, bis(3,5-di-tert-butyl-4-hydroxybenzyl)sulfide, isooctyl-3,5-di-tert-butyl-4-hydroxybenzylmercaptoacetate.

5 1.8. Hydroxybenzylated malonates, for example dioctadecyl-2,2-bis(3,5-di-tert-butyl-2-hydroxybenzyl)malonate, di-octadecyl-2-(3-tert-butyl-4-hydroxy-5-methylbenzyl)malonate, didodecylmercaptoethyl-2,2-bis-(3,5-di-tert-butyl-4-hydroxybenzyl)malonate, bis[4-(1,1,3,3-tetramethylbutyl)phenyl]-2,2-bis(3,5-di-tert-butyl-4-hydroxybenzyl)malonate.

10 1.9. Aromatic hydroxybenzyl compounds, for example 1,3,5-tris(3,5-di-tert-butyl-4-hydroxybenzyl)-2,4,6-trimethylbenzene, 1,4-bis(3,5-di-tert-butyl-4-hydroxybenzyl)-2,3,5,6-tetramethylbenzene, 2,4,6-tris(3,5-di-tert-butyl-4-hydroxybenzyl)phenol.

15 1.10. Triazine compounds, for example 2,4-bis(octylmercapto)-6-(3,5-di-tert-butyl-4-hydroxyanilino)-1,3,5-triazine, 2-octylmercapto-4,6-bis(3,5-di-tert-butyl-4-hydroxyanilino)-1,3,5-triazine, 2-octylmercapto-4,6-bis(3,5-di-tert-butyl-4-hydroxyphenoxy)-1,3,5-triazine, 2,4,6-tris(3,5-di-tert-butyl-4-hydroxyphenoxy)-1,2,3-triazine, 1,3,5-tris-(3,5-di-tert-butyl-4-hydroxybenzyl)isocyanurate, 1,3,5-tris(4-tert-butyl-3-hydroxy-2,6-dimethylbenzyl)isocyanurate, 2,4,6-tris(3,5-di-tert-butyl-4-hydroxyphenylethyl)-1,3,5-triazine, 1,3,5-tris(3,5-di-tert-butyl-4-hydroxyphenylpropionyl)-hexahydro-1,3,5-triazine, 1,3,5-tris(3,5-dicyclohexyl-4-hydroxybenzyl)isocyanurate.
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1.11. Acylaminophenols, for example 4-hydroxylauranilide, 4-hydroxystearanilide, octyl N-(3,5-di-tert-butyl-4-hydroxyphenyl)carbamate.

25 1.12. Esters of β -(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid with mono- or polyhydric alcohols, e.g. with methanol, ethanol, n-octanol, i-octanol, octadecanol, 1,6-hexanediol, 1,9-nonanediol, ethylene glycol, 1,2-propanediol, neopentyl glycol, thiodiethylene glycol, diethylene glycol, triethylene glycol, pentaerythritol, tris(hydroxyethyl) isocyanurate, N,N'-bis(hydroxyethyl)oxamide, 3-thiaundecanol, 3-thiapentadecanol, trimethylhexanediol, trimethylolpropane, 4-hydroxymethyl-1-phospha-2,6,7-trioxabicyclo[2.2.2]octane.
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1.13. Esters of β -(5-tert-butyl-4-hydroxy-3-methylphenyl)propionic acid with mono- or polyhydric alcohols, e.g. with methanol, ethanol, n-octanol, i-octanol, octadecanol, 1,6-hexanediol, 1,9-nonanediol, ethylene glycol, 1,2-propanediol, neopentyl glycol, thiodiethylene glycol,

diethylene glycol, triethylene glycol, pentaerythritol, tris(hydroxyethyl) isocyanurate, N,N'-bis-(hydroxyethyl)oxamide, 3-thiaundecanol, 3-thiapentadecanol, trimethylhexanediol, trimethylolpropane, 4-hydroxymethyl-1-phospha-2,6,7-trioxabicyclo[2.2.2]octane.

- 5 1.14. Esters of β -(3,5-dicyclohexyl-4-hydroxyphenyl)propionic acid with mono- or polyhydric alcohols, e.g. with methanol, ethanol, octanol, octadecanol, 1,6-hexanediol, 1,9-nonanediol, ethylene glycol, 1,2-propanediol, neopentyl glycol, thiodiethylene glycol, diethylene glycol, triethylene glycol, pentaerythritol, tris(hydroxyethyl)isocyanurate, N,N'-bis(hydroxyethyl)oxamide, 3-thiaundecanol, 3-thiapentadecanol, trimethylhexanediol, trimethylolpropane, 4-hydroxymethyl-1-phospha-2,6,7-trioxabicyclo[2.2.2]octane.

- 15 1.15. Esters of 3,5-di-tert-butyl-4-hydroxyphenyl acetic acid with mono- or polyhydric alcohols, e.g. with methanol, ethanol, octanol, octadecanol, 1,6-hexanediol, 1,9-nonanediol, ethylene glycol, 1,2-propanediol, neopentyl glycol, thiodiethylene glycol, diethylene glycol, triethylene glycol, pentaerythritol, tris(hydroxyethyl)isocyanurate, N,N'-bis(hydroxyethyl)oxamide, 3-thiaundecanol, 3-thiapentadecanol, trimethylhexanediol, trimethylolpropane, 4-hydroxymethyl-1-phospha-2,6,7-trioxabicyclo[2.2.2]octane.

- 20 1.16. Amides of β -(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid e.g. N,N'-bis(3,5-di-tert-butyl-4-hydroxyphenylpropionyl)hexamethylenediamide, N,N'-bis(3,5-di-tert-butyl-4-hydroxyphenylpropionyl)trimethylenediamide, N,N'-bis(3,5-di-tert-butyl-4-hydroxyphenylpropionyl)hydrazide, N,N'-bis[2-(3-[3,5-di-tert-butyl-4-hydroxyphenyl]propionyloxy)ethyl]oxamide (Nau-gard®XL-1 supplied by Uniroyal).

- 25 1.17. Ascorbic acid (vitamin C)

- 30 1.18. Aminic antioxidants, for example N,N'-di-isopropyl-p-phenylenediamine, N,N'-di-sec-butyl-p-phenylenediamine, N,N'-bis(1,4-dimethylpentyl)-p-phenylenediamine, N,N'-bis(1-ethyl-3-methylpentyl)-p-phenylenediamine, N,N'-bis(1-methylheptyl)-p-phenylenediamine, N,N'-dicyclohexyl-p-phenylenediamine, N,N'-diphenyl-p-phenylenediamine, N,N'-bis(2-naphthyl)-p-phenylenediamine, N-isopropyl-N'-phenyl-p-phenylenediamine, N-(1,3-dimethylbutyl)-N'-phenyl-p-phenylenediamine, N-(1-methylheptyl)-N'-phenyl-p-phenylenediamine, N-cyclohexyl-N'-phenyl-p-phenylenediamine, 4-(p-toluenesulfamoyl)diphenylamine, N,N'-dimethyl-N,N'-di-sec-butyl-p-phenylenediamine, diphenylamine, N-allyldiphenylamine, 4-isopropoxydiphenylamine, 1,1-phenyl-1-naphthylamine, N-(4-tert-butylphenyl)-1-

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2. UV absorbers and light stabilisers

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tyl-5'-(2-methoxycarbonylethyl)-2'-hydroxyphenyl]-2H-benzotriazole with polyethylene glycol 300; $\left[R-CH_2CH_2-COO-CH_2CH_2 \right]_2$ where R = 3'-tert-butyl-4'-hydroxy-5'-2H-benzotriazol-2-ylphenyl, 2-[2'-hydroxy-3'-(α,α -dimethylbenzyl)-5'-(1,1,3,3-tetramethylbutyl)phenyl]-benzotriazole; 2-[2'-hydroxy-3'-(1,1,3,3-tetramethylbutyl)-5'-(α,α -dimethylbenzyl)phenyl]benzotriazole.

2.2. 2-Hydroxybenzophenones, for example the 4-hydroxy, 4-methoxy, 4-octyloxy, 4-decyloxy, 4-dodecyloxy, 4-benzyloxy, 4,2',4'-trihydroxy and 2'-hydroxy-4,4'-dimethoxy derivatives.

2.3. Esters of substituted and unsubstituted benzoic acids, as for example 4-tertbutyl-phenyl salicylate, phenyl salicylate, octylphenyl salicylate, dibenzoyl resorcinol, bis(4-tert-butylbenzoyl)resorcinol, benzoyl resorcinol, 2,4-di-tert-butylphenyl 3,5-di-tert-butyl-4-hydroxybenzoate, hexadecyl 3,5-di-tert-butyl-4-hydroxybenzoate, octadecyl 3,5-di-tert-butyl-4-hydroxybenzoate, 2-methyl-4,6-di-tert-butylphenyl 3,5-di-tert-butyl-4-hydroxybenzoate.

2.4. Acrylates, for example ethyl α -cyano- β,β -diphenylacrylate, isooctyl α -cyano- β,β -diphenylacrylate, methyl α -carbomethoxycinnamate, methyl α -cyano- β -methyl-p-methoxycinnamate, butyl α -cyano- β -methyl-p-methoxycinnamate, methyl α -carbomethoxy-p-methoxycinnamate and N-(β -carbomethoxy- β -cyanovinyl)-2-methylindoline.

2.5. Nickel compounds, for example nickel complexes of 2,2'-thio-bis-[4-(1,1,3,3-tetramethylbutyl)phenol], such as the 1:1 or 1:2 complex, with or without additional ligands such as n-butylamine, triethanolamine or N-cyclohexyldiethanolamine, nickel dibutyldithiocarbamate, nickel salts of the monoalkyl esters, e.g. the methyl or ethyl ester, of 4-hydroxy-3,5-di-tert-butylbenzylphosphonic acid, nickel complexes of ketoximes, e.g. of 2-hydroxy-4-methylphenyl undecylketoxime, nickel complexes of 1-phenyl-4-lauroyl-5-hydroxypyrazole, with or without additional ligands.

2.6. Sterically hindered amines, for example bis(2,2,6,6-tetramethyl-4-piperidyl)sebacate, bis(2,2,6,6-tetramethyl-4-piperidyl)succinate, bis(1,2,2,6,6-pentamethyl-4-piperidyl)sebacate, bis(1-octyloxy-2,2,6,6-tetramethyl-4-piperidyl)sebacate, bis(1,2,2,6,6-pentamethyl-4-piperidyl) n-butyl-3,5-di-tert-butyl-4-hydroxybenzylmalonate, the condensates of 1-(2-hydroxyethyl)-2,2,6,6-tetramethyl-4-piperidine and succinic acid, linear or cyclic condensates of

N,N'-bis(2,2,6,6-tetramethyl-4-piperidyl)hexamethylenediamine and 4-tert-octylamino-2,6-dichloro-1,3,5-triazine, tris(2,2,6,6-tetramethyl-4-piperidyl)nitrilotriacetate, tetrakis(2,2,6,6-tetramethyl-4-piperidyl)-1,2,3,4-butanetetracarboxylate, 1,1'-(1,2-ethanediyl)-bis(3,3,5,5-tetramethylpiperazinone), 4-benzoyl-2,2,6,6-tetramethylpiperidine, 4-stearyloxy-2,2,6,6-tetramethylpiperidine, bis(1,2,2,6,6-pentamethylpiperidyl)-2-n-butyl-2-(2-hydroxy-3,5-di-tert-butylbenzyl)-malonate, 3-n-octyl-7,7,9,9-tetramethyl-1,3,8-triazaspiro[4.5]decane-2,4-dione, bis(1-octyloxy-2,2,6,6-tetramethylpiperidyl)sebacate, bis(1-octyloxy-2,2,6,6-tetramethylpiperidyl)succinate, linear or cyclic condensates of N,N'-bis(2,2,6,6-tetramethyl-4-piperidyl)hexamethylenediamine and 4-morpholino-2,6-dichloro-1,3,5-triazine, the condensate of 2-chloro-4,6-bis(4-n-butylamino-2,2,6,6-tetramethylpiperidyl)-1,3,5-triazine and 1,2-bis(3-aminopropylamino)ethane, the condensate of 2-chloro-4,6-di-(4-n-butylamino-1,2,2,6,6-pentamethylpiperidyl)-1,3,5-triazine and 1,2-bis(3-aminopropylamino)ethane, 8-acetyl-3-dodecyl-7,7,9,9-tetramethyl-1,3,8-triazaspiro[4.5]decane-2,4-dione, 3-dodecyl-1-(2,2,6,6-tetramethyl-4-piperidyl)pyrrolidin-2,5-dione, 3-dodecyl-1-(1,2,2,6,6-pentamethyl-4-piperidyl)pyrrolidine-2,5-dione, a mixture of 4-hexadecyloxy- and 4-stearyloxy-2,2,6,6-tetramethylpiperidine, a condensation product of N,N'-bis(2,2,6,6-tetramethyl-4-piperidyl)hexamethylenediamine and 4-cyclohexylamino-2,6-dichloro-1,3,5-triazine, a condensation product of 1,2-bis(3-aminopropylamino)ethane and 2,4,6-trichloro-1,3,5-triazine as well as 4-butylamino-2,2,6,6-tetramethylpiperidine (CAS Reg. No. [136504-96-6]); N-(2,2,6,6-tetramethyl-4-piperidyl)-n-dodecylsuccinimid, N-(1,2,2,6,6-pentamethyl-4-piperidyl)-n-dodecylsuccinimid, 2-undecyl-7,7,9,9-tetramethyl-1-oxa-3,8-diaza-4-oxo-spiro[4,5]decane, a reaction product of 7,7,9,9-tetramethyl-2-cycloundecyl-1-oxa-3,8-diaza-4-oxospiro [4,5]decane und epichlorohydrin, 1,1-bis(1,2,2,6,6-pentamethyl-4-piperidyl)oxycarbonyl-2-(4-methoxyphenyl)ethene, N,N'-bis-formyl-N,N'-bis(2,2,6,6-tetramethyl-4-piperidyl)hexamethylenediamine, diester of 4-methoxy-methylenemalonic acid with 1,2,2,6,6-pentamethyl-4-hydroxypiperidine, poly[methylpropyl-3-oxy-4-(2,2,6,6-tetramethyl-4-piperidyl)]siloxane, reaction product of maleic acid anhydride- α -olefin-copolymer with 2,2,6,6-tetramethyl-4-aminopiperidine or 1,2,2,6,6-pentamethyl-4-aminopiperidine.

2.7. Oxamides, for example 4,4'-dioctyloxyoxanilide, 2,2'-diethoxyoxanilide, 2,2'-dioctyloxy-5,5'-di-tert-butoxanilide, 2,2'-didodecyloxy-5,5'-di-tert-butoxanilide, 2-ethoxy-2'-ethyloxanilide, N,N'-bis(3-dimethylaminopropyl)oxamide, 2-ethoxy-5-tert-butyl-2'-ethoxanilide and its mixture with 2-ethoxy-2'-ethyl-5,4'-di-tert-butoxanilide, mixtures of o- and p-methoxy-disubstituted oxanilides and mixtures of o- and p-ethoxy-disubstituted oxanilides.

2.8. 2-(2-Hydroxyphenyl)-1,3,5-triazines, for example 2,4,6-tris(2-hydroxy-4-octyloxyphenyl)-1,3,5-triazine, 2-(2-hydroxy-4-octyloxyphenyl)-4,6-bis(2,4-dimethylphenyl)-1,3,5-triazine, 2-(2,4-dihydroxyphenyl)-4,6-bis(2,4-dimethylphenyl)-1,3,5-triazine, 2,4-bis(2-hydroxy-4-propyloxyphenyl)-6-(2,4-dimethylphenyl)-1,3,5-triazine, 2-(2-hydroxy-4-octyloxyphenyl)-4,6-bis(4-methylphenyl)-1,3,5-triazine, 2-(2-hydroxy-4-dodecyloxyphenyl)-4,6-bis(2,4-dimethylphenyl)-1,3,5-triazine, 2-(2-hydroxy-4-tridecyloxyphenyl)-4,6-bis(2,4-dimethylphenyl)-1,3,5-triazine, 2-[2-hydroxy-4-(2-hydroxy-3-butyloxy-propoxy)phenyl]-4,6-bis(2,4-dimethyl)-1,3,5-triazine, 2-[2-hydroxy-4-(2-hydroxy-3-octyloxy-propyloxy)phenyl]-4,6-bis(2,4-dimethyl)-1,3,5-triazine, 2-[4-(dodecyloxy/tridecyloxy-2-hydroxypropoxy)-2-hydroxy-phenyl]-4,6-bis(2,4-dimethylphenyl)-1,3,5-triazine, 2-[2-hydroxy-4-(2-hydroxy-3-dodecyloxypropoxy)phenyl]-4,6-bis(2,4-dimethylphenyl)-1,3,5-triazine, 2-(2-hydroxy-4-hexyloxy)phenyl-4,6-diphenyl-1,3,5-triazine, 2-(2-hydroxy-4-methoxyphenyl)-4,6-diphenyl-1,3,5-triazine, 2,4,6-tris[2-hydroxy-4-(3-butoxy-2-hydroxypropoxy)phenyl]-1,3,5-triazine, 2-(2-hydroxyphenyl)-4-(4-methoxyphenyl)-6-phenyl-1,3,5-triazine, 2-[2-hydroxy-4-[3-(2-ethylhexyl-1-oxy)-2-hydroxypropyloxy]phenyl]-4,6-bis(2,4-dimethylphenyl)-1,3,5-triazine.

3. Metal deactivators, for example N,N'-diphenyloxamide, N-salicylal-N'-salicyloyl hydrazine, N,N'-bis(salicyloyl) hydrazine, N,N'-bis(3,5-di-tert-butyl-4-hydroxyphenylpropionyl) hydrazine, 3-salicyloylamino-1,2,4-triazole, bis(benzylidene)oxalyl dihydrazide, oxanilide, isophthaloyl dihydrazide, sebacoyl bisphenylhydrazide, N,N'-diacetyl adipoyl dihydrazide, N,N'-bis(salicyloyl)oxalyl dihydrazide, N,N'-bis(salicyloyl)thiopropionyl dihydrazide.

4. Hydroxylamines, for example, N,N-dibenzylhydroxylamine, N,N-diethylhydroxylamine, N,N-dioctylhydroxylamine, N,N-dilaurylhydroxylamine, N,N-ditetradecylhydroxylamine, N,N-dihexadecylhydroxylamine, N,N-dioctadecylhydroxylamine, N-hexadecyl-N-octadecylhydroxylamine, N-heptadecyl-N-octadecylhydroxylamine, N,N-dialkylhydroxylamine derived from hydrogenated tallow amine.

5. Nitrones, for example, N-benzyl-alpha-phenylnitron, N-ethyl-alpha-methylnitron, N-octyl-alpha-heptylnitron, N-lauryl-alpha-undecylnitron, N-tetradecyl-alpha-tridecylnitron, N-hexadecyl-alpha-pentadecylnitron, N-octadecyl-alpha-heptadecylnitron, N-hexadecyl-alpha-heptadecylnitron, N-octadecyl-alpha-pentadecylnitron, N-heptadecyl-alpha-heptadecyl-

nitron, N-octadecyl-alpha-hexadecylnitron, nitron derived from N,N-dialkylhydroxylamine derived from hydrogenated tallow amine.

6. Thiosynergists, for example dilauryl thiodipropionate or distearyl thiodipropionate.

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7. Peroxide scavengers, for example esters of β -thiodipropionic acid, for example the lauryl, stearyl, myristyl or tridecyl esters, mercaptobenzimidazole or the zinc salt of 2-mercaptobenzimidazole, zinc dibutyldithiocarbamate, dioctadecyl disulfide, pentaerythritol tetrakis(β -dodecylmercapto)propionate.

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8. Polyamide stabilisers, for example copper salts in combination with iodides and/or phosphorus compounds and salts of divalent manganese.

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9. Basic co-stabilisers, for example, melamine, polyvinylpyrrolidone, dicyandiamide, triallyl cyanurate, urea derivatives, hydrazine derivatives, amines, polyamides, polyurethanes, alkali metal salts and alkaline earth metal salts of higher fatty acids, for example calcium stearate, zinc stearate, magnesium behenate, magnesium stearate, sodium ricinoleate and potassium palmitate, antimony pyrocatecholate or zinc pyrocatecholate.

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10. Nucleating agents, for example, inorganic substances such as talcum, metal oxides such as titanium dioxide or magnesium oxide, phosphates, carbonates or sulfates of, preferably, alkaline earth metals; organic compounds such as mono- or polycarboxylic acids and the salts thereof, e.g. 4-tert-butylbenzoic acid, adipic acid, diphenylacetic acid, sodium succinate or sodium benzoate; polymeric compounds such as ionic copolymers (ionomers).

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11. Fillers and reinforcing agents, for example calcium carbonate, silicates, glass fibres, glass bulbs, asbestos, talc, kaolin, mica, barium sulfate, metal oxides and hydroxides, carbon black, graphite, wood flour and flours or fibers of other natural products, synthetic fibers.

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12. Other additives, for example, plasticisers, lubricants, emulsifiers, pigments, rheology additives, catalysts, flow control agents, optical brighteners, flameproofing agents, antistatic agents and blowing agents.

The following examples illustrate the invention.

Analytical procedures:

Intrinsic Viscosity (I.V.):

- 5 1g polymer is dissolved in 100g of a mixture of phenol/di-chloro-benzene (1/1). The viscosity of this solution is measured at 30°C in an Ubelode-viscosimeter and recalculated to the intrinsic viscosity.

Color:

- 10 Color (b value of the color difference formula) is measured according to ASTM D1925. using a Hunter Lab Scan spectrometer.

Melt Flow Rate (MFR):

MFR is determined within Goettfert MP-P according to ISO 1133.

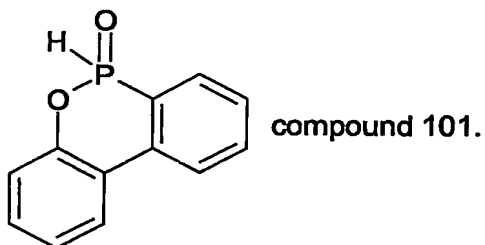
15 Materials:

PET: Polyclear T94 from KoSa Gersthofen

Allinco® from DSM (Dodeca hydro-1,1'-carbonyl-bis-azepin-2-one, CAS RN 19494-73-6)

- 20 IRGAMOD 195 (from Ciba Specialty Chemicals)
IRGAFOS 12 (from Ciba Specialty Chemicals)
IRGAFOS 168 (from Ciba Specialty Chemicals)
IRGANOX HP136 (from Ciba Specialty Chemicals)

- 25 Compound 101 synthesized according to standard procedures



PET melt processing (extrusion):

General procedure:

In a twin screw extruder (ZSK 25 from Werner & Pfleiderer) with screws rotating in the same direction, the below mentioned formulations are extruded at a temperature of $T_{\max} = 280^{\circ}\text{C}$ (heating zone 1 - 6), a throughput of 5 kg/h and 100 rev/min and pelletised in a water bath.

5 Comparative Example2 C1-C3:

The general procedure is applied to a composition of 100% Polyclear T94, 0.1% Irgamod 195 and 0.3% Allinco. The results are given in Table 1.

Table 1: Comparative examples

	Additive	MFR [g/10min]	I.V. dl/g	Pellet color B*-value
Comp. 1	Extrusion without additives	16	0.74	1.5
Comp. 1	0.3% Allinco	13	0.82	5.3
Comp. 2	0.1% Irgamod 195	14	0.79	2.1

10 Inventive Examples 1 to 5:

The general procedure is repeated with the only difference that the compounds listed in Table 2 are added. The results are given in Table 2.

Table 2

Ex. No.	Additive	MFR	I.V. dl/g	Pellet color B*-value
1	0.3% Allinco + 0.1% Irgamod 195	14	0.81	-0.2
2	0.3% Allinco + 0.1% Irgafos 12	11	0.86	n.d.
3	0.3% Allinco + 0.1% Irgafos 168	11	0.83	n.d.
4	0.3% Allinco + 0.1% Irganox HP136	9.7	0.90	n.d.
5	0.3% Allinco + 0.1% compound 101	15	0.77	-0.9

n.d.: not determined

Claims

1. A process for increasing the molecular weight and/or for the modification of a polycondensate, which process comprises adding to the polycondensate

a) at least one bis-acyllactam;

b1) at least one phosphite, phosphinate or phosphonate; or

b2) at least one benzofuran-2-one type compound or

b3) at least one phosphite, phosphinate or phosphonate and one benzofuran-2-one type compound

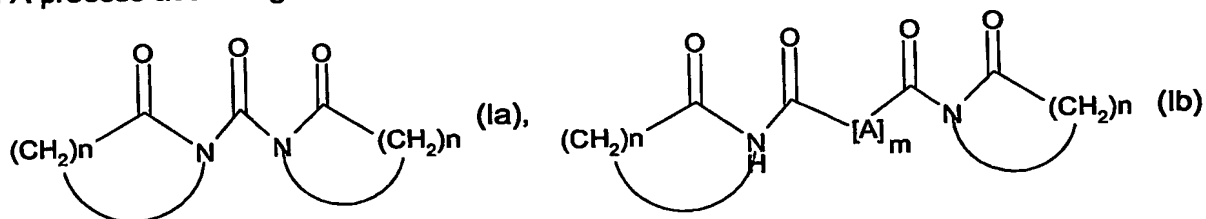
and processing the mixture in the melt.

2. A process according to claim 1, wherein the polycondensate is an aliphatic or aromatic polyester, an aliphatic or aromatic polyamide or polycarbonate, or a blend or copolymer thereof.

3. A process according to claim 1, wherein the polycondensate is polyethylene terephthalate (PET), polybutylene terephthalate (PBT), polyethyleneterephthalate (PEN), a copolyester, PA 6, PA 6,6, a polycarbonate containing bisphenol A, bisphenol Z or bisphenol F linked via carbonate groups.

4. A process according to claim 1, wherein the polycondensate is PET, a copolymer with PET or PBT.

5. A process according to claim 1 wherein the bis-acyllactam is of formula Ia or Ib

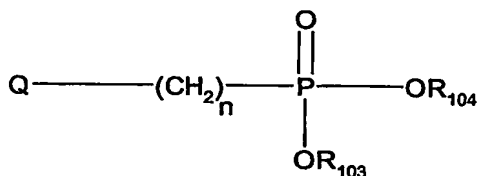


wherein A is C_1 - C_{18} alkylen, C_2 - C_{18} alkylene interrupted by at least one oxygen atom, C_1 - C_{18} alkenylene, phenylene, phenylene- C_1 - C_{18} alkylene, C_1 - C_{18} alkylene-phenylene, or C_1 - C_{18} alkylene-phenylene- C_1 - C_{18} alkylene;

m is 0 or 1 and

n is a number from 3 to 12.

6. A process according to claim 1, wherein the phosphonate is of formula II



(II), wherein

R_{103} is H, $\text{C}_1\text{-C}_{20}$ alkyl, unsubstituted or $\text{C}_1\text{-C}_4$ alkyl-substituted phenyl or naphthyl,

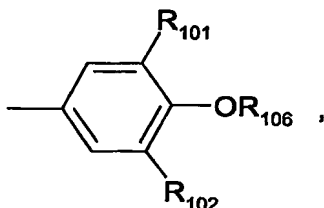
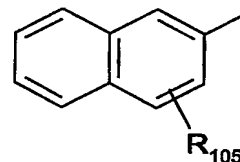
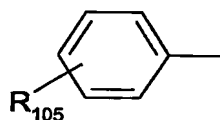
5 R_{104} is hydrogen, $\text{C}_1\text{-C}_{20}$ alkyl, unsubstituted or $\text{C}_1\text{-C}_4$ alkyl-substituted phenyl or naphthyl; or M^{r+} / r,

M^{r+} is an r-valent metal cation or the ammonium ion,

n is 0, 1, 2, 3, 4, 5 or 6, and

r is 1, 2, 3 or 4;

10 Q is hydrogen, $-\text{X}-\text{C}(\text{O})-\text{OR}_{107}$, or a radical



R_{101} is isopropyl, tert-butyl, cyclohexyl, or cyclohexyl which is substituted by 1-3 $\text{C}_1\text{-C}_4$ alkyl groups,

15 R_{102} is hydrogen, $\text{C}_1\text{-C}_4$ alkyl, cyclohexyl, or cyclohexyl which is substituted by 1-3 $\text{C}_1\text{-C}_4$ alkyl groups,

R_{105} is H, $\text{C}_1\text{-C}_{18}$ alkyl, OH, halogen or $\text{C}_3\text{-C}_7$ cycloalkyl;

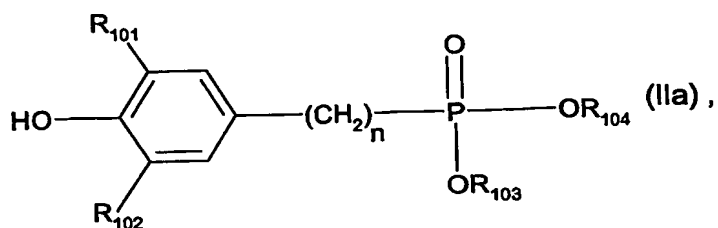
R_{106} is H, methyl, trimethylsilyl, benzyl, phenyl, sulfonyl or $\text{C}_1\text{-C}_{18}$ alkyl;

R_{107} is H, $\text{C}_1\text{-C}_{10}$ alkyl or $\text{C}_3\text{-C}_7$ cycloalkyl; and

X is phenylene, $\text{C}_1\text{-C}_4$ alkyl group-substituted phenylene or cyclohexylene.

20

7. A process according to claim 6 wherein the phosphonate is of formula IIa



wherein

R₁₀₁ is H, isopropyl, tert-butyl, cyclohexyl, or cyclohexyl which is substituted by 1-3 C₁-C₄alkyl groups,

R₁₀₂ is hydrogen, C₁-C₄alkyl, cyclohexyl, or cyclohexyl which is substituted by 1-3 C₁-C₄alkyl groups,

R₁₀₃ is C₁-C₂₀alkyl, unsubstituted or C₁-C₄alkyl-substituted phenyl or naphthyl,

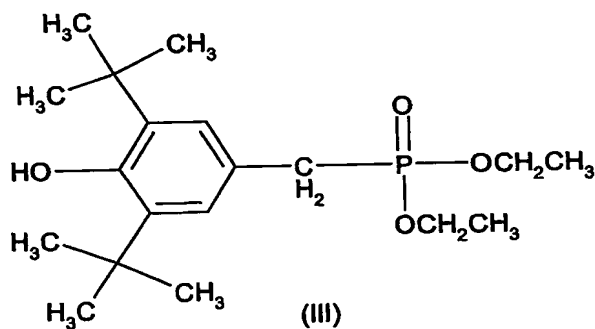
R₁₀₄ is hydrogen, C₁-C₂₀alkyl, unsubstituted or C₁-C₄alkyl-substituted phenyl or naphthyl; or

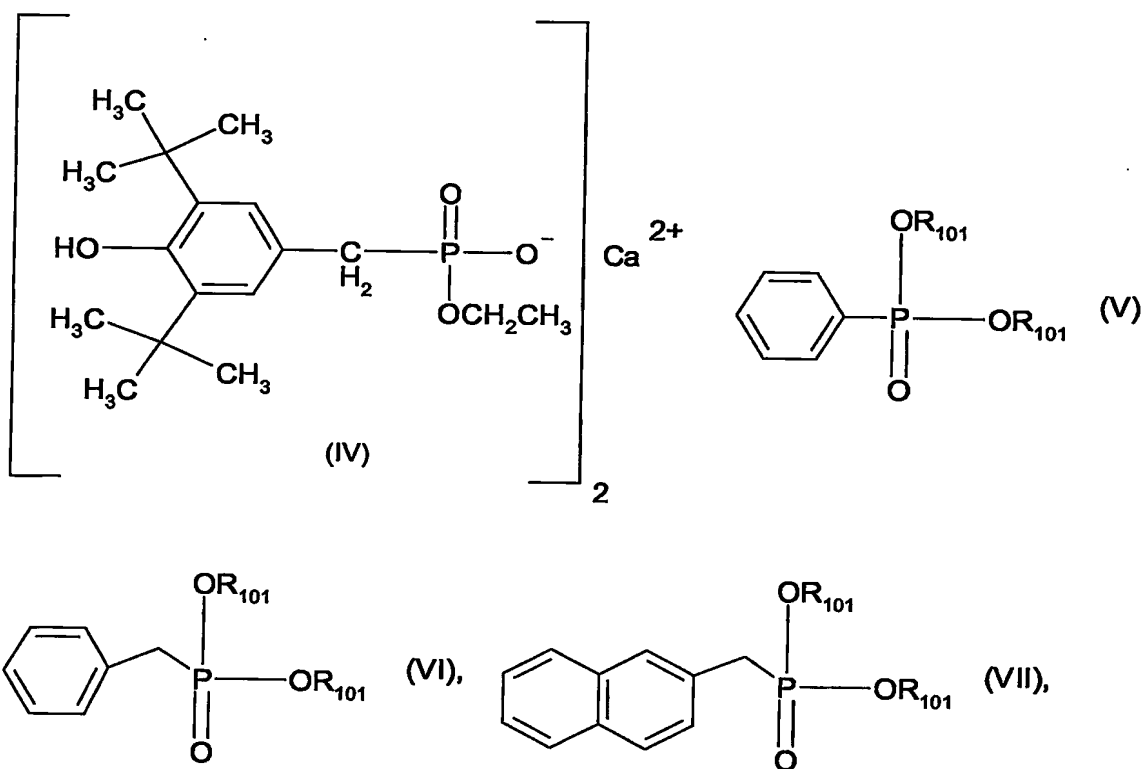
M^{r+} / r,

M^{r+} is an r-valent metal cation, r is 1, 2, 3 or 4; and

n is 1, 2, 3, 4, 5 or 6.

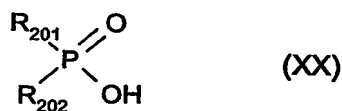
8. A process according to claim 1, wherein the phosphonate is of formula III, IV, V, VI or VII :





wherein the R_{101} are each independently of one another hydrogen or M^{r+} / r.;

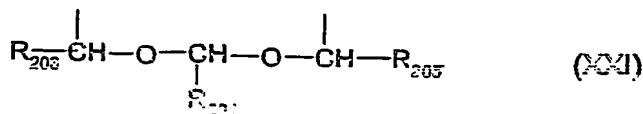
9. A process according to claim 1 wherein the phosphinates are of the formula XX



wherein

R_{201} is hydrogen, C_1 - C_{20} alkyl, phenyl or C_1 - C_4 alkyl substituted phenyl; biphenyl, naphthyl, $-CH_2-O-C_1-C_{20}$ alkyl or $-CH_2-S-C_1-C_{20}$ alkyl,

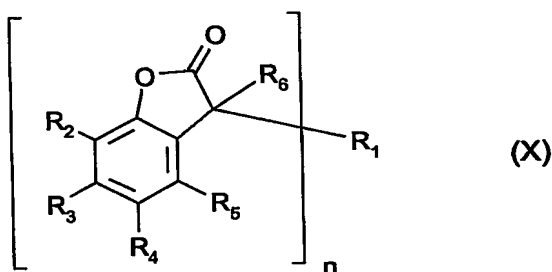
R_{202} is C_1 - C_{20} alkyl, phenyl or C_1 - C_4 alkyl substituted phenyl; biphenyl, naphthyl, $-CH_2-O-C_1-C_{20}$ alkyl or $-CH_2-S-C_1-C_{20}$ alkyl, or R_1 and R_2 together are a radical of the formula XXI



wherein

R_{203} , R_{204} and R_{205} independently of each other are C_1 - C_{20} alkyl, phenyl or C_1 - C_4 alkyl substituted phenyl.

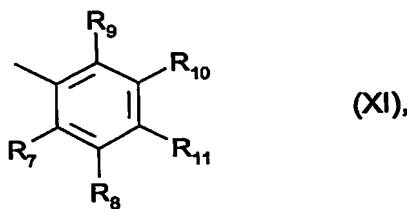
- 5 10. A process according to claim 1, wherein the benzofuran-2-one type compound is of formula X



- 10 wherein, if $n = 1$,

R_1 is naphthyl, phenanthryl, anthryl, 5,6,7,8-tetrahydro-2-naphthyl, 5,6,7,8-tetrahydro-1-naphthyl, thienyl, benzo[b]thienyl, naphtho[2,3-b]thienyl, thianthrenyl, dibenzofuryl, chromenyl, xanthenyl, phenoxathiinyl, pyrrolyl, imidazolyl, pyrazolyl, pyrazinyl, pyrimidinyl, pyridazinyl, indoliziny, isoindolyl, indolyl, indazolyl, purinyl, quinoliziny, isoquinolyl, quinolyl, phthalazinyl, naphthyridinyl, quinoxaliny, quinazolinyl, cinnoliny, pteridinyl, carbazolyl, β -carboliny, phenanthridinyl, acridinyl, perimidiny, phenanthrolinyl, phenazinyl, isothiazolyl, phenothiazinyl, isoxazolyl, furazanyl, biphenyl, terphenyl, fluorenyl or phenoxazinyl, each of which is unsubstituted or substituted by C_1 - C_4 alkyl, C_1 - C_4 alkoxy, C_1 - C_4 alkylthio, hydroxy, halogen, amino, C_1 - C_4 alkylamino, phenylamino or di(C_1 - C_4 -alkyl)amino, or R_1 is a radical of

- 20 formula XI



and,

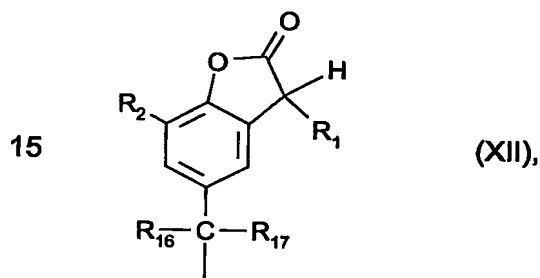
if $n = 2$,

R_1 is unsubstituted or C_1 - C_4 alkyl- or hydroxy-substituted phenylene or naphthylene; or $-R_{12}-X-R_{13}-$,

- 5 R_2, R_3, R_4 and R_5 are each independently of one another hydrogen, chloro, hydroxy, C_1 - C_{25} -alkyl, C_7 - C_9 phenylalkyl, unsubstituted or C_1 - C_4 alkyl-substituted phenyl; unsubstituted or C_1 - C_4 alkyl-substituted C_5 - C_8 cycloalkyl; C_1 - C_{18} alkoxy, C_1 - C_{18} alkylthio, C_1 - C_4 alkylamino, di(C_1 - C_4 -alkyl)amino, C_1 - C_{25} alkanoyloxy, C_1 - C_{25} alkanoylamino, C_3 - C_{25} alkenoyloxy;

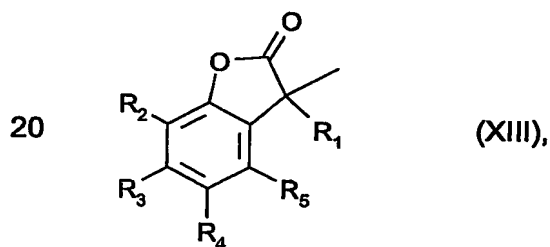
C_3 - C_{25} alkanoyloxy which is interrupted by oxygen, sulfur or $\text{>N}-R_{14}$; C_6 - C_9 cycloalkylcar-

- 10 bonyloxy, benzoyloxy or C_1 - C_{12} alkyl-substituted benzoyloxy; or R_2 and R_3 , or R_3 and R_4 , or R_4 and R_5 , together with the linking carbon atoms, form a benzene ring, R_4 is additionally $-(CH_2)_p-COR_{15}$ or $-(CH_2)_qOH$ or, if R_3, R_5 and R_6 are hydrogen, R_4 is additionally a radical of formula XII



wherein R_1 is as defined above for $n = 1$,

R_6 is hydrogen or a radical of formula XIII



wherein R_1 is not a radical of formula XII, and R_1 is as defined above for $n = 1$

R_7, R_8, R_9, R_{10} and R_{11} are each independently of one another hydrogen, halogen, hydroxy,

C_1-C_{25} alkyl; C_2-C_{25} alkyl which is interrupted by oxygen, sulfur or $\text{>N}-R_{14}$; C_1-C_{25} alkoxy;

C_2-C_{25} alkoxy which is interrupted by oxygen, sulfur or $\text{>N}-R_{14}$; C_1-C_{25} alkylthio, C_3-C_{25} -

- 5 alkenyl, C_3-C_{25} alkenyloxy, C_3-C_{25} alkynyl, C_3-C_{25} alkynyloxy, C_7-C_9 phenylalkyl, C_7-C_9 phenyl-alkoxy, unsubstituted or C_1-C_4 alkyl-substituted phenyl; unsubstituted or C_1-C_4 alkyl-substituted phenoxy; unsubstituted or C_1-C_4 alkyl-substituted C_5-C_8 cycloalkyl; unsubstituted or C_1-C_4 alkyl-substituted C_5-C_8 cycloalkoxy; C_1-C_4 alkylamino, di(C_1-C_4 alkyl)amino,

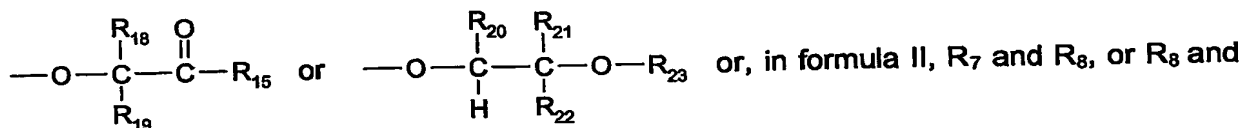
C_1-C_{25} alkanoyl; C_3-C_{25} alkanoyl which is interrupted by oxygen, sulfur or $\text{>N}-R_{14}$;

C_1-C_{25} alkanoyloxy; C_3-C_{25} alkanoyloxy which is interrupted by oxygen, sulfur or $\text{>N}-R_{14}$;

- 10 C_1-C_{25} alkanoylamino, C_3-C_{25} alkenoyl; C_3-C_{25} alkenoyl which is interrupted by oxygen, sulfur or $\text{>N}-R_{14}$; C_3-C_{25} alkenoyloxy; C_3-C_{25} alkenoyloxy which is interrupted by oxygen, sulfur

or $\text{>N}-R_{14}$; C_6-C_9 cycloalkylcarbonyl, C_6-C_9 cycloalkylcarbonyloxy, benzoyl or

C_1-C_{12} alkyl-substituted benzoyl; benzoyloxy or C_1-C_{12} alkyl-substituted benzoyloxy;



- 15 R_{11} , together with the linking carbon atoms, form a benzene ring,
 R_{12} and R_{13} are each independently of the other unsubstituted or C_1-C_4 alkyl-substituted phenylene or naphthylene,
 R_{14} is hydrogen or C_1-C_8 alkyl,

R_{15} is hydroxy, $\left[-O^- \frac{1}{r} M^{r+} \right]$, C_1-C_{18} alkoxy or $\text{>N} \begin{array}{l} R_{24} \\ R_{25} \end{array}$,

R_{16} and R_{17} are each independently of the other hydrogen, CF_3 , C_1 - C_{12} alkyl or phenyl, or R_{16} and R_{17} , together with the linking carbon atom, are a C_5 - C_8 cycloalkylidene ring which is unsubstituted or substituted by 1 to 3 C_1 - C_4 alkyl;

R_{18} and R_{19} are each independently of the other hydrogen, C_1 - C_4 alkyl or phenyl,

5 R_{20} is hydrogen or C_1 - C_4 alkyl,

R_{21} is hydrogen, unsubstituted or C_1 - C_4 alkyl-substituted phenyl; C_1 - C_{25} alkyl; C_2 - C_{25} alkyl

which is interrupted by oxygen, sulfur or $\text{>N}-R_{14}$; C_7 - C_9 phenylalkyl which is unsubstituted or substituted at the phenyl moiety by 1 to 3 C_1 - C_4 alkyl; C_7 - C_{25} phenylalkyl which is

interrupted by oxygen, sulfur or $\text{>N}-R_{14}$ and which is unsubstituted or substituted at the

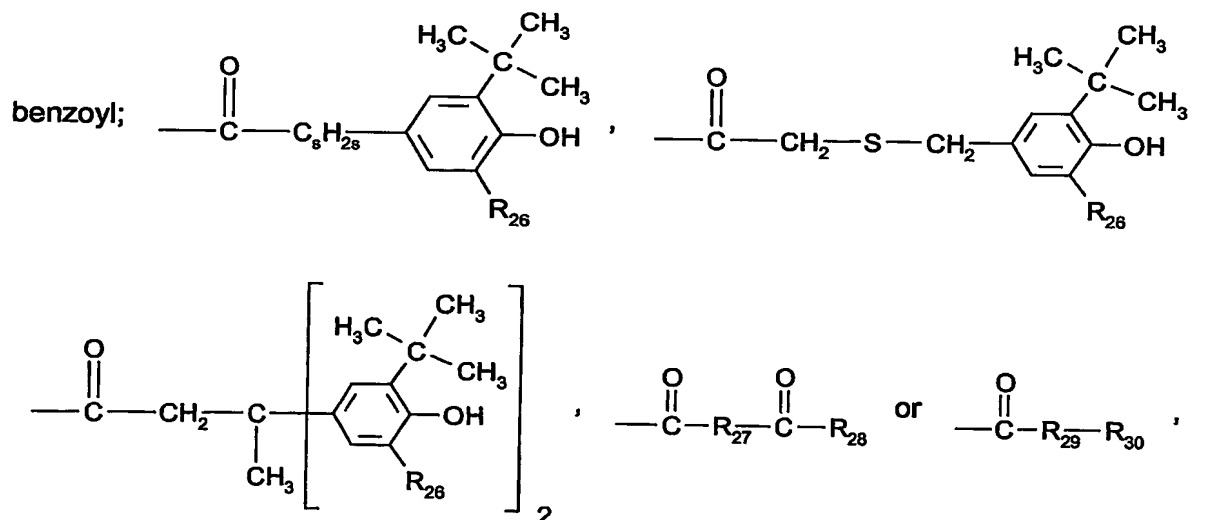
10 phenyl moiety by 1 to 3 C_1 - C_4 alkyl, or R_{20} and R_{21} , together with the linking carbon atoms, form a C_5 - C_{12} cycloalkylene ring which is unsubstituted or substituted by 1 to 3 C_1 - C_4 alkyl;

R_{22} is hydrogen or C_1 - C_4 alkyl,

R_{23} is hydrogen, C_1 - C_{25} alkanoyl, C_3 - C_{25} alkenoyl; C_3 - C_{25} alkanoyl which is interrupted by

oxygen, sulfur or $\text{>N}-R_{14}$; C_2 - C_{25} alkanoyl which is substituted by a di(C_1 - C_6 alkyl)phos-

15 phonate group; C_6 - C_9 cycloalkylcarbonyl, thenoyl, furoyl, benzoyl or C_1 - C_{12} alkyl-substituted

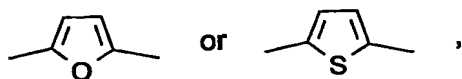


R_{24} and R_{25} are each independently of the other hydrogen or C_1 - C_{18} alkyl,

R_{26} is hydrogen or C_1 - C_6 alkyl,

R_{27} is a direct bond, C_1 - C_{18} alkylene; C_2 - C_{18} alkylene which is interrupted by oxygen, sulfur or

$\text{N}-R_{14}$; C_2 - C_{18} alkenylene, C_2 - C_{20} alkylidene, C_7 - C_{20} phenylalkylidene, C_5 - C_8 cycloalkylene, C_7 - C_8 bicycloalkylene, unsubstituted or C_1 - C_4 alkyl-substituted phenylene,



5 R_{28} is hydroxy, $\left[-O^- \frac{1}{r} M^{r+} \right]$, C_1 - C_{18} alkoxy or $-N \begin{matrix} R_{24} \\ R_{25} \end{matrix}$,

R_{29} is oxygen, -NH- or $\text{N} \begin{matrix} \text{O} \\ \parallel \\ -C- \end{matrix} \text{NH}-R_{30}$,

R_{30} is C_1 - C_{18} alkyl or phenyl,

R_{31} is hydrogen or C_1 - C_{18} alkyl,

M is an r-valent metal cation,

10 X is a direct bond, oxygen, sulfur or $-NR_{31}-$,

n is 1 or 2,

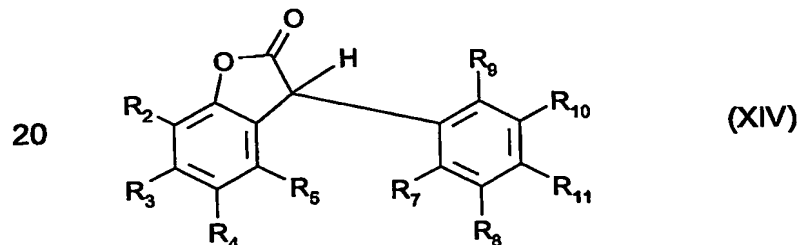
p is 0, 1 or 2,

q is 1, 2, 3, 4, 5 or 6,

r is 1, 2 or 3, and

15 s is 0, 1 or 2.

11. A process according to claim 10, wherein the benzofuran-2-one type compound is of formula XIV



wherein

R₂ is hydrogen or C₁-C₆alkyl,

R₃ is hydrogen,

R₄ is hydrogen or C₁-C₆alkyl,

R₅ is hydrogen,

- 5 R₇, R₈, R₉, R₁₀ and R₁₁ are each independently of one another hydrogen, C₁-C₄alkyl, C₁-C₄-

alkoxy or $\text{---O---}\begin{array}{c} \text{R}_{20} \quad \text{R}_{21} \\ | \quad | \\ \text{C} \text{---} \text{C} \text{---} \text{O---R}_{23} \\ | \quad | \\ \text{H} \quad \text{R}_{22} \end{array}$, with the proviso that at least two of R₇, R₈, R₉, R₁₀ or

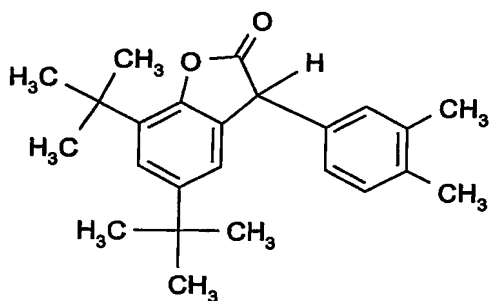
R₁₁ are hydrogen,

R₂₀, R₂₁ and R₂₃ are hydrogen, and

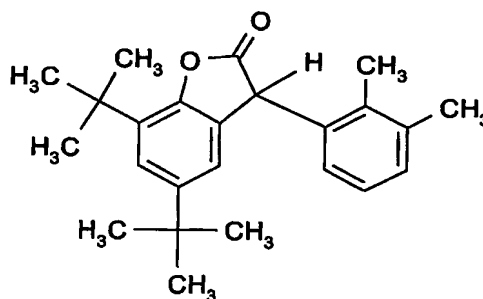
R₂₃ is C₂-C₄alkanoyl.

10

12. A process according to claim 11, wherein the benzofuran-2-one type compound is of formula XIVa or XIVb



(XIVa)

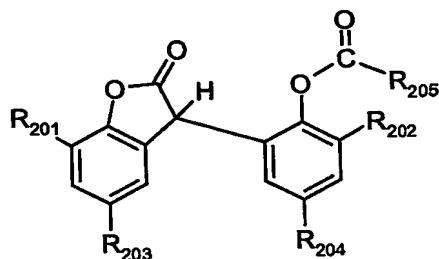


(XIVb)

15

or a mixture or blend of the two compounds of formulae XIVa and XIVb.

13. A process according to claim 1, wherein the benzofuran-2-one type compound is of formula XV



(XV)

wherein

R_{201} and R_{202} are each independently of one another hydrogen or C_1 - C_8 alkyl,

5 R_{203} and R_{204} are each independently of one another C_1 - C_{12} alkyl, and

R_{205} is C_1 - C_7 alkyl.

14. A process according to claim 1, wherein the bis-acyllactam is used in an amount of 0.01 to 5 % by weight based on the weight of the polycondensate.

10 15. A process according to claim 1, wherein the phosphite, phosphinate or phosphonate is used in an amount of 0.01 to 5 % by weight based on the weight of the polycondensate.

15 16. A process according to claim 1, wherein the benzofuran-2-one type compound is used in an amount of 0.01 to 5 % by weight based on the weight of the polycondensate.

17. A process according to claim 1, wherein the ratio of the bis-acyllactam to the phosphite, phosphinate, phosphonate or to the benzofuran-2-one type compound or the sum of all is from 1:10 to 5:1.

18. A process according to claim 1, wherein the maximum mass-temperature of the melt is from 170° to 320° C.

19. A composition comprising

25 a) a polycondensate;

b) at least one bis-acyllactam;

c1) at least one phosphite, phosphinate or phosphonate; or

c2) at least one benzofuran-2-one type compound or

30 c3) at least one phosphite, phosphinate or phosphonate and one benzofuran-2-one type compound.

20. A polycondensate obtainable by a process according to claim 1.

21. Use of a mixture of

- 5 a) at least one bis-acyllactam;
- b1) at least one phosphite, phosphinate or phosphonate; or
- b2) at least one benzofuran-2-one type compound or
- b3) at least one phosphite, phosphinate or phosphonate and one benzofuran-2-one type compound
- 10 for increasing the molecular weight and/or for the modification of a polycondensate.

Abstract

The present invention relates to a process for increasing the molecular weight and for the modification of polycondensates, to the use of an additive blend effecting the increase in molecular weight without imparting color to the polycondensates as well as to the polycondensates obtainable by that process. The additive blend contains at least one bis-acyllactam, at least one phosphite, phosphinate or phosphonate; or at least one benzofuran-2-one type compound or at least one phosphite, phosphinate or phosphonate and one benzofuran-2-one type compound.

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